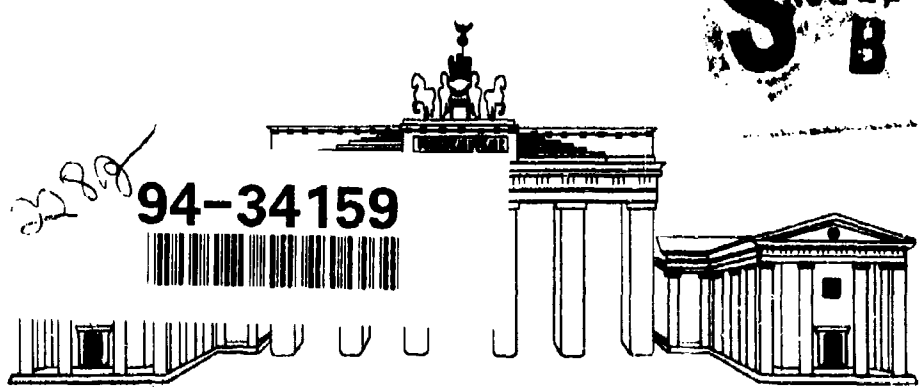
X-Ray Topography and High Resolution Diffraction

PROGRAMME AND ABSTRACTS



Humboldt-University
and Max-Planck-Arbeitsgruppe "Röntgenbeugung"

Berlin, Germany, 5-7 September 1994

2nd European Symposium

X-Ray Topography and High Resolution Diffraction

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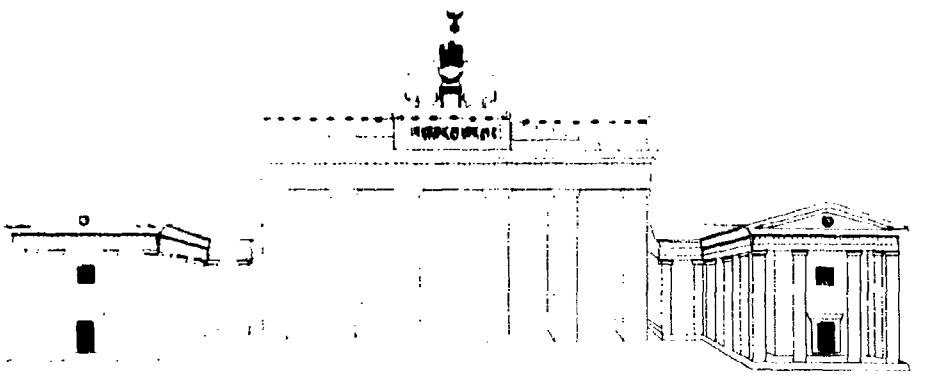

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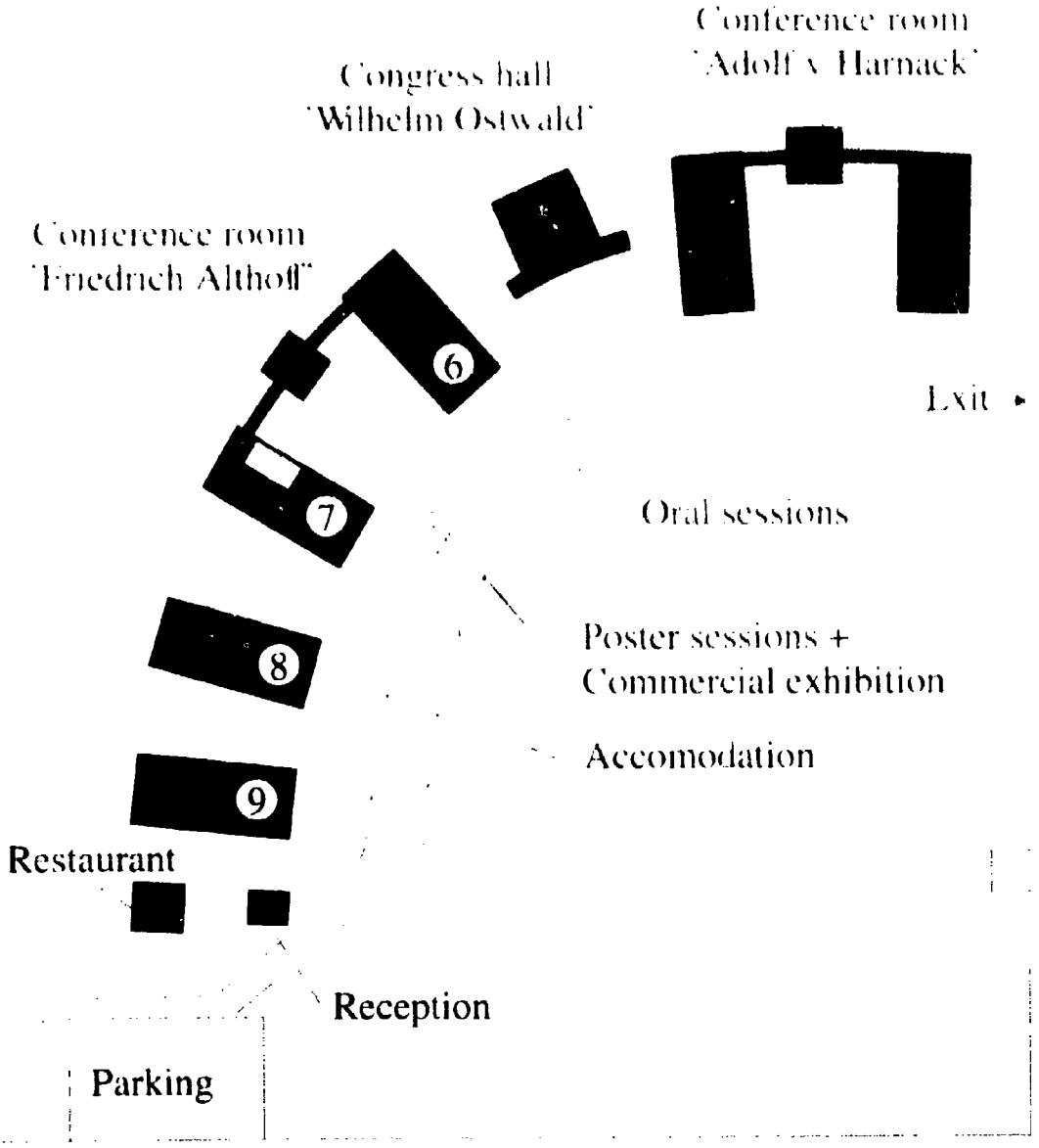

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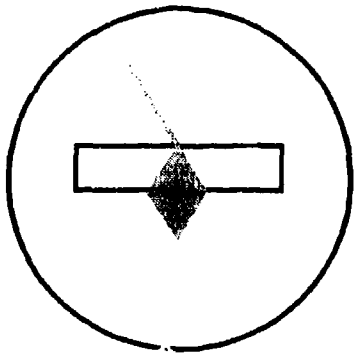

2nd European Symposium

X-Ray Topography and High Resolution Diffraction

Berlin, Germany, 5-7 September 1994

PROGRAMME

Monday 5th September

09.00 **H. Klapper** ***Opening***

09.10 **G. Hildebrandt** (invited)
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Topography in magnetism: domain and phase transition investigations p. 8

10.30 ***Coffee Break***

11.00 **J. Baruchel, P.Rejmankova** (invited)
Diffraction topographic investigations of crystals under applied electric fields p. 9

11.25 **I. L. Smolsky** (invited)
Dependence of defect arrangement in crystals on surface morphology and on conditions of growth from solution p. 10

11.45 **M. Polcarova, A.George, J.Bradler, A.Jacques, J.Gemperlova** (invited)
Interaction of slip bands with grain boundaries - observation by white beam SR topography p. 11

12.10 **S.J. Barnett, A.M.Keir, A.G.Cullis, A.D.Johnson** (invited)
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14.40	**P. Kidd, P.F.Fewster, N.L.Andrew** Interpretation of the diffraction profile resulting from strain relaxation in epilayers	(invited) p. 15
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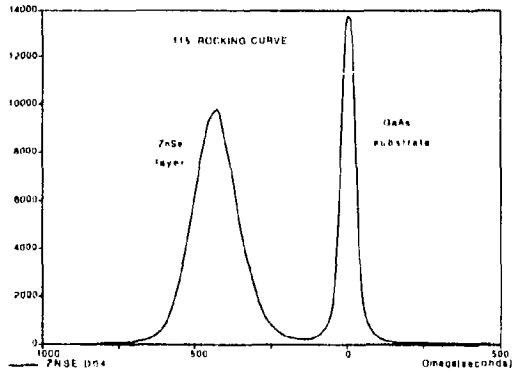

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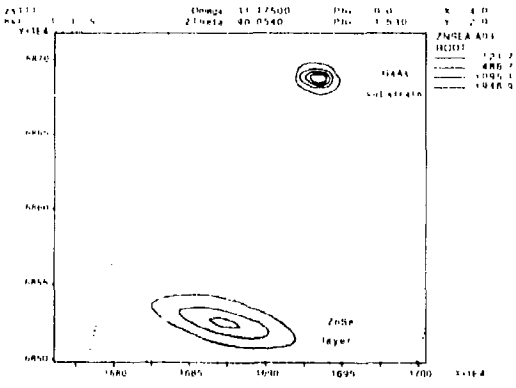

high resolution triple axis mode

Figures are 115 CuKa reflections from a ZnSe on (001) GaAs sample provided by Dr Michael Heuken, University of Aachen.

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EARLY EXPERIMENTAL PROOFS OF THE DYNAMICAL THEORY

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Darwin's dynamical theory (which was restricted to reflected X-rays) strongly disagreed with his experiments (1914), but measurements during the next twenty years of half widths of the reflection curve, integrated reflection etc., mostly on calcite, confirmed the theory more and more. The general theory (Ewald 1917, Laue 1931), first applied to higher order Bragg angles, was then able to interpret deficiency lines (Wagner 1920) and Umweganregung (Renninger 1937) as three waves interactions in the Bragg case. After some unconscious confirmations of the theory in the Laue case (Cork, Murdock, Tahvonen 1932 - 1935) and the first evaluation of Pendellösung fringes (McGillavry 1940), it was mainly the discovery of new properties of interfering X-rays in thick quartz and calcite single crystals (anomalously low absorption, particular direction of energy propagation) by G. Borrmann (1941/50) that initiated expansions of the theory and provided a deeper insight into Ewald's wave-fields. Later experiments mainly with silicon crystals by Authier, Bonse, Kato, Lang and many others (on beam paths, Pendellösung, topography, interferometry etc.) expanded the field to a veritable "Kristalloptik der Röntgenstrahlen" as it was called already in 1917 by P.P. Ewald.

Gerhard Borrmann (born 1908) about 1968. This photograph was taken by P.P. Ewald in the English Garden in Munich where he inspired Laue to perform his diffraction experiment, early in 1912.

TOPOGRAPHY IN MAGNETISM: DOMAIN AND PHASE TRANSITION INVESTIGATIONS

Michel Schlenker[1] **and Jose Baruchel**[2]

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[2] *European Synchrotron Radiation Facility, BP 220, F-38043 Grenoble*

Modern diffraction imaging started with the development of Lang's techniques of section and traverse X-ray topography. The pioneering and indeed much of the foundation work in the use of topography in magnetism was that of Polcarova, who, in a series of papers starting in 1962 [1], analyzed the magnetostrictive distortion due to ferromagnetic domains and how it produces contrast on X ray images.

Magnetism has used both X ray topography, which has produced by far the larger number of works, with an ever increasing part for synchrotron radiation (SR), and neutron topography. They have complementary capabilities for the investigation both of magnetic domains and of phase coexistence.

The salient advantage of neutrons [2] is that they interact *directly* with the magnetic structure. Local changes in the magnetic structure, i.e. magnetic domains, can give rise to changes in reflectivity and be imaged, even if they do not correspond to changes in macroscopic magnetization nor in lattice distortion. Thus antiferromagnetic domains of various kinds, including spin density wave domains in chromium [3], chirality (handedness) domains in helimagnets and 180° (time-reversed) domains in rutile-type transition metal fluorides, could be explored and some aspects of their behavior investigated for the first time. In phase coexistence observation, neutrons offer the possibility of directly characterizing the different phases. However, this technique is slow and has very poor resolution because there are few neutrons. Furthermore it was inactive for three years due to the shutdown of Institut Laue Langevin. It will resume in 1995, and cooperation can be discussed.

X-ray topography in the form used till now, i.e. with Thomson (electron charge) scattering, relies on the changes in distortion associated with domains or different phases. Thus practically all first-order phase transitions [4], but only some kinds of domains, can be observed. The speed and good resolution of synchrotron radiation make it convenient to observe domain and phase boundary movements under the effect of temperature and magnetic field variations [2,4]. Very valuable results were also obtained on the physics of the magnetic materials that host the domains or walls, e.g. the strain distribution due to local ion implantation in garnets [5].

Modern synchrotron radiation sources, with their high intensity beams (a factor of 10^2 larger than on standard SR sources) including high energy photons, and small sources at large distances from the specimen, are fine tools for topography, among others in magnetism. In particular the resolution (spatial or angular), can be tailored to some extent and is good. The possibility of topographic investigations using magnetic X-ray scattering is exciting.

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DIFFRACTION TOPOGRAPHIC INVESTIGATIONS OF CRYSTALS UNDER AN APPLIED ELECTRIC FIELD

J. Baruchel and P.Rejmánkova
European Synchrotron Radiation Facility, BP 220, 38043 Grenoble, France

Diffraction topography has been used to investigate the effect of an applied dc field on single crystals which exhibit ionic conductivity and piezoelectricity. Results were reported in quartz [1-3], KH_2PO_4 [4], $KTiOPO_4$ [5], $LiN_2H_5SO_4$ and $LiNH_4SO_4$ [6]...and α-$LiIO_3$ [7-10]. Similar studies were carried out on other crystals like Si [11].

A summary of these electric field (from $\approx 10^2$ to 10^6 Vm^{-1}) related effects is not a simple task because their magnitude is temperature and sample dependent, and their occurrence is a function of the applied field range, and of the time delay after the field application. The experimental facts which are listed below are consequently not always observed simultaneously on a given experiment:
1) dense population of fine lines running along the ionic conductivity axis; the details of these lines can be a function of the electrode actual setting
2) visibility of some of growth bands and other defects, which were nearly invisible at zero field
3) occurrence of nearly planar defects parallel to the ionic conductivity axis and to the main surfaces of the platelet shaped crystals
4) enhanced gradient of distortion in the neighbourhood of some of the surfaces
5) thicker lines associated with a migration of ions from the electrode into the crystal
6) non visibility of some of the defects present at zero field

Examples of these points, obtained either using classical sources or synchrotron radiation, for different materials will be presented. The influence of the nature and irregularities of the electrodes will be discussed. We will emphasize on the new possibilities of 'section' topography at modern facilities like the ESRF.

A non uniform nearly one-dimensional ionic conductance is mainly invoked as a basis for the interpretation of the observed effects: the current mostly flows either through "channels" or crystal inhomogeneities, or in the neighbourhood of surfaces, leading to the occurrence of a non homogeneous space charge density.

But several questions require more work to find a definite answer. The most important one concerns the mechanism connecting the variation of space charge to the observed gradients of distortion. It seems probable that the piezoelectric properties of these compounds, which display the same symmetry as the observed effects [10], are to be taken into account when working out this mechanism in more detail.

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DEPENDENCE OF DEFECT ARRANGEMENT IN CRYSTALS ON SURFACE MORPHOLOGY AND ON CONDITIONS OF GROWTH FROM SOLUTION

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X-ray topographic methods have been used for studies of real structure of crystals grown from solution (KDP group) in relation with growth conditions.

Systematic deviations of dislocations from the minimum-energy orientations [1] have been observed. These deviations are step-like at crystallisation temperatures lower 35°C and more smooth at higher temperatures. They are related with influence of the morphology of the growing face, predominantly macrosteps, on the orientations of the grown-in dislocations. The alternation of the macrosteps and the groups of elemental growth steps can bring to significant deviation of the dislocation direction from the minimum energy orientation. Nevertheless there is no contradiction with the theory [1].

Diffusion processes take place during growth from solutions. They affect on the crystal defects and therefore can be fixed. X-ray topographs show helices or dislocation tangles formed on the initially screw dislocations as a result of inhomogeneity in impurity distribution or intrinsic point defects near the macrostep edges. Activity of the diffusion processes increases with increasing supersaturation. Distribution of defects and inhomogeneities are usually forming by the seed geometry and regeneration conditions. Using of "point seed" allows to decrease inner strains in the crystal and to control the crystal habit in correspondence with the dislocation growth mechanism.

Crystals grown from solutions with different pH meanings show difference in the crystals homogeneity. Contrast of the growth bands and sectorial boundaries is almost invisible on the X-ray topographs made from crystals grown at the pH 2.3 but it is strong in crystals grown at the pH = 4.8 in the same regime. Connection of this effect with the difference in the distribution ratio for tri- and divalent cationic impurities is supposed.

Reference

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INTERACTION OF SLIP BANDS WITH GRAIN BOUNDARIES – OBSERVATION BY WHITE BEAM SR TOPOGRAPHY

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The influence of grain boundary (GB) type on transmission of slip bands was studied by X-ray diffraction topography. Observation was performed on bicrystals of Fe-Si alloys (4 and 6at%Si), the GBs were special ones with low Σ. First experiments were made using laboratory X-ray sources and several topographic techniques on specimens deformed by compression. The topographic contrast was evaluated quantitatively by comparison with simulated images. The calculation was based on a simple model of dislocation pile-ups at GBs [1]. The SR *in situ* experiments were performed at LURE, Orsay, using white beam reflection topography. The specimens were situated in a tensile stage produced at Ecole des Mines de Nancy. The topographic images of samples under tension were observed using X-ray sensitive camera. Major changes in the slip band pattern were recorded on photographic plates.

The ease of slip transmission across GB depends on the parameter $\mathbf{M} = (\mathbf{L}_1 \cdot \mathbf{L}_2) \cdot (\mathbf{b}_1 \cdot \mathbf{b}_2)$, where $\mathbf{L}_i$ are directions of intersection lines of slip planes with GB and $\mathbf{b}_i$ are slip directions in the two grains. The higher its value, the easier the transmission of slip. Besides this geometric criterion the stresses exerted by dislocation pile-ups in one grain are important for slip system formation in the second grain [2]. Three different types of samples with different values of the parameter **M** have been chosen. Nucleation and propagation of slip bands, their stop at the GB and transmission across it were observed. Unloading of the specimen did not cause any change of the slip bands. The plastic deformation developed usually independently in both grains, the transmission of slip bands across the grain boundary was observed rarely even in the cases of maximal value **M**=1 (the Burgers vectors and slip planes parallel in both grains). Two explanations of this effect are suggested. The GB structure itself influences significantly the slip transmission. The real GBs differ slightly from the theoretical small Σ misorientation, therefore several systems of dislocations appear in the GBs which interact with the slip dislocations. The GB planes are favourite sites for impurity segregation and a variation of Si concentration. The atomic structure of GB is modified and thus the barrier for the dislocation movement is strengthened.

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IN-SITU X-RAY TOPOGRAPHY STUDIES DURING THE MBE GROWTH PROCESS OF InGaAs STRAINED LAYERS

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We report recent results from a novel facility constructed to enable in-situ X-ray diffraction studies during the MBE growth of III-V strained layer device structures on 50mm diameter substrates[1]. This new facility, used in conjunction with the Daresbury synchrotron source, permits X-ray topographic (XRT) imaging of individual misfit dislocations formed during the MBE growth process. The misfit dislocation growth and interactions can be imaged as a function of layer thickness, strain, growth and post-growth conditions.

Recent results have shown that the nature and distribution of dislocations threading up from the substrate are crucial in determining the initial pattern of epilayer relaxation. Under certain growth conditions and substrate dislocation distributions large areas of the epilayer remain free of misfit dislocations at epilayer thicknesss significantly higher than the measured initial critical thickness t_{1c}. We have observed, in-situ for the first time, a second critical thickness t_{2c} (under certain conditions $t_{2c} > 2t_{1c}$[2]) at which there is a rapid increase in misfit dislocation density as a second dislocation source becomes active. The precise nature of this second source will be identified by detailed TEM analysis on areas of sample accurately targeted by the in-situ XRT.

The XRT Experiments are described and the demanding experimental requirements imposed by the in-situ nature of these experiments are discussed. Recent experimental results demonstrating newly revealed aspects of the relaxation process are presented.

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Double- and Triple-Crystal X-Ray Diffraction Analysis of Semiconductor Quantum Wires

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This paper describes the potentialities of high-resolution x-ray diffractometry to investigate and characterize nondestructively the geometrical and structural properties of corrugated surfaces (surface gratings) and periodic quantum wires. The periodically corrugated surfaces were prepared by holographic lithography and subsequent reactive ion etching of (100)GaAs surfaces, while the quantum wires were fabricated by using InAs/GaAs quantum wells grown by molecular beam epitaxy onto (100)GaAs substrates.[1,2]

The surface gratings and quantum wires are investigated by using double- and triple-crystal x-ray diffraction measurements and x-ray reciprocal space mapping. The experimental patterns are analyzed by using a semidynamical diffraction model. The dynamical theory is used in order to describe the x-ray diffraction of the substrate crystal, while the kinematical theory is used to describe the x-ray diffraction of the surface grating. The interference between different parts of the periodic surface array is described by using the multiple-slit Fraunhofer formalism. The simulation of the experimental diffraction patterns allows us to determine the shape and the geometrical parameters (period, width, height, etc.) of the surface corrugations and quantum wires.[3]

By measuring reflection curves in the symmetrical and asymmetrical diffraction geometries we found a partial elastic strain relaxation of such quantum structures for which the unit cell was tetragonally distorted before the etching process. The strain relaxation results in an orthorhombic deformation of the unit cell.[2,4] The elastic strain relaxation phenomenon in quantum wires can be explained and described quantitatively by using Hook's law and applying appropriate boundary conditions at the wire-substrate interface.[4] In our strain model, we assume that the lattice coherence at the wire-substrate interface is achieved only along the wire direction, due to the limited lateral extension of the wires.

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Applications in Multiple Crystal Diffractometry

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The elucidation of the structure of semiconductor multilayers can be adequately determined by x-ray methods but the interpretation is not always straight-forward. The presence of defects and diffraction effects can create significant changes to the diffraction pattern that requires a more complete analysis by diffraction space mapping. Interpreting the diffraction space maps when obtained with the High Resolution Muliple-Crystal Multiple-Reflection Diffractometer permits the use of three extra very powerful tools. The first is the use of multiple-crystal topography to relate diffraction space intensity features to lateral contrast on the photographic emulsion, the use of accurate lattice parameter determination of diffraction features and the simulation of the diffraction shapes using dynamical theory.

Examples of these procedures will be presented in this talk.

INTERPRETATION OF THE DIFFRACTION PROFILE RESULTING FROM STRAIN RELAXATION IN EPILAYERS

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With the advent of high resolution triple axis diffractometry and its application to reciprocal space mapping, we are now in a position to make quantitative measurements of the distributions of crystal strains and rotations arising from arrays of defects in crystals. When a strained epitaxial layer relaxes, the interfacial misfit dislocations are rarely ideal infinitely long or regularly spaced edge dislocations. Thus, in addition to the displacements required for the relief of misfit, the dislocation strainfields and threading arms cause local bending and tilting of the crystal planes. Relief of misfit strain is measured as a shift in the layer Bragg peak position, whereas local bending and tilting of the crystal planes in the vicinity of dislocations gives rise to diffuse scatter around the Bragg peak. The aim of this work is to interpret and model the diffraction profiles in terms of the underlying dislocation distributions in the relaxing layers.

We have studied the profiles of the Bragg peaks and diffuse scatter in reciprocal space along both the plane perpendicular ($q_\perp$) and plane parallel ($q_\parallel$) directions for sample structures consisting of layers of $In_xGa_{1-x}As$ grown by MBE on [001] oriented GaAs substrates. The samples have different layer thicknesses and different dislocation distributions. We have measured the dislocation distributions in the interfaces using plan view TEM. We find that for thin layers with low dislocation densities the diffraction profiles in both the plane perpendicular ($q_\perp$) and plane parallel ($q_\parallel$) can be modelled by considering two components of the diffraction profile, namely; dynamical scattering from the coherently coupled regions of perfect layer between dislocations and diffuse scatter from decoupled regions around the dislocations. From the $q_\parallel$ profile a lateral dimension can be associated with the regions which give rise to the diffuse scatter, and we show that this dimension scales with the layer thickness. For thicker layers with higher dislocation densities, the strainfields of the dislocations overlap. In this case the diffraction profiles in ($q_\perp$) are modelled by considering the ratio of the depth of coherently scattering decoupled crystal, above the dislocation array, with the total depth of the layer, assuming that scattering from the greatly distorted crystal close to the array is lost. Along $q_\parallel$ the diffuse scatter is modelled on the basis of a statistical distribution of finite correlation lengths and microscopic tilts.

Study of thin buried interfaces in III-V compound heterostructures by high resolution X-ray diffraction

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Interface sharpness and planarity are of fundamental importance in determining electrical and optical properties of devices based on extremely thin active regions. To study this problem high resolution x-ray diffraction techniques can be used in two different ways:

i) in multi quantum well based heterostructures the interface parameters can be determined by fitting the superlattice peak intensity and broadening in x-ray rocking curves.

ii) in single quantum well heterostructures the intensity fringes arising from the of x-ray standing wave interference between substrate and cap layer depends on the well thickness and mismatch.

As an example of the first case, the InGaAs to InP and InP to InGaAs interfaces in InP based structures are studied. It is shown that heavy incorporation of As up to 20% in the InP and P in the InGaAs growth surfaces occur due to surface exposure to the competing group V atom flux (1). From the rocking curve analysis incorporation depth and local As or P concentration are evaluated.

As for the second point is concerned, the interface sharpness in InGaAs/GaAs based heterostructures is known to be severely affected by the In segregation toward the growth surface (2). From literature In segregation length values ranging from 1 to several monolayers are reported. In this work a new approach based on the measurements of x-ray diffraction profiles of single monolayer InAs/GaAs structures in different geometrical conditions is developed. A segregation length of nearly 2 monolayers, slightly dependent on the growth conditions is found.

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TEXTURE EVOLUTION IN THIN FILMS AND MULTILAYERS VIA SYNCHROTRON TOPOGRAPHY AND GIXS:

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Sputter deposited films and multilayers are used for a wide variety of applications including protective coatings on turbine engine blades, magnetic recording heads, optical elements, electronic packaging, x-ray filters and monochromator components to name a few examples. This wide range of interest requires growth thicknesses from a few nanometers to tens of microns depending on the particular product. In many applications control, or even possible manipulation, of the growth texture is essential for producing the desired physical properties. Fundamental knowledge of the growth texture in the growth direction, as well as within the plane of growth, as a function of processing conditions is necessary. No single technique is capable of scanning the entire thickness range of film growth. A variety of x-ray methods including grazing incidence x-ray scattering, conventional pole figure studies and white beam synchrotron topography were used to study texture evolution for the thinnest films up to the thickest multilayer coatings. The results of these texture measurements are discussed in terms of the evolving microstructure observed via transmission electron microscopy.

X-RAY DOUBLE CRYSTAL TECHNIQUE AS A TOOL FOR POINT LIKE DEFECT STUDY

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X-ray experiments like multi-crystal diffraction techniques are usually seen as tools for studying large lattice imperfections and probes for significant crystal volumes.But even with beam sizes around $1mm^2$ there is a wealth of information to be gained by appropriate design and execution of the experiment. Point like defects always have been on the list of potential subjects for the study by X-ray techniques but either the size or density of the defects had to be on the scale acceptable by X-ray experiment. In case of crystal defects whose size is smaller then the resolving power of the X-ray method some averaged values of the diffracted intensity can only be measured and this being subject to diffuse scattering experiments. By applying *in-situ* controlled conditions of the experiment like: temperature, illumination, pressure, magnetic field, electric field etc. it is possible in some cases to acquire additional information regarding point like defects. For the first time X-ray experiment was able to successfully record and follow transition of metastable EL2 defects in SI GaAs [1]. El2 being already recognized as AsGa (arsenic antisite) single atom defect is a prime example of the potential of X-ray diffraction methods. Double crystal diffraction technique used in this case confirmed results and predictions from optical absorption measurements of this material as well as it yielded some new unexpected information as to the behavior of the EL2. This new approach was then applied to study DX center in AlGaAs:Te [2] as well as to examine novel Low Temperature GaAs MBE layers [3,4]. Especially in the LTGaAs results of measurements are spectacular in a way that they lead to and confirm that in this case EL2-like defects are divided into two groups with different time constants for their metastable behavior [5]

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SURFACE AND INTERFACE SENSITIVE X-RAY SCATTERING

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The sensitivity of X-ray scattering to the state of surface and/or interfaces in layered samples substantially depends on the penetration depth of X-rays. Very small penetration depth can be achieved in the following experimental modes:

- extremely asymmetric coplanar diffraction (AXRD),
- non coplanar grazing incidence diffraction (GID), and
- X-ray reflection (i.e. diffraction with zero diffraction vector – XRR).

X-ray scattering in perfect structures (flat interfaces, no volume defects) can be described by means of the dynamical diffraction theory. In all the above modes, interface defects (roughness) reduce the coherently scattered intensity (diffuse extinction) and increases the divergence of the scattered beams (diffuse X-ray scattering). Theoretical description of these processes is based on the Distorted-Wave-Born-Approximation (DWBA). In this method, the diffuse scattering is described as a transition between two undisturbed dynamical wavefields in the perfect structure.

The scattered intensity can be represented being the function of the wave vector transfer $\mathbf{Q} = \mathbf{K}_{out} - \mathbf{K}_{in}$. In the reciprocal space distribution of the scattered intensity several characteristic features can be observed. The correlation properties of the interface roughness concentrate the diffusely scattered intensity distribution into small areas close to the reciprocal lattice points. The shape of these maxima depends both on in-plane and on vertical (inter-plane) roughness correlations. Dynamical scattering processes are the cause of resonant peaks (the Yoneda wings in XRR, for instance) that are stretched along the Ewald spheres in reciprocal space. All these effects can be observed by X-ray diffractometry and reflectometry that enables us to scan the scattered intensity distribution in reciprocal plane near the reciprocal lattice point.

In spite of the fact that all the experimental modes can be described in an uniform way, they characterize the interfaces from different viewpoints. XRR is sensitive only to interface morphology, while the diffraction methods (AXRD and GID) also to crystalline quality. GID is extremely sensitive to small in-plane atomar shifts and, therefore, to lateral strain relaxation in layers.

In the lecture, both theoretical and experimental aspects will be reviewed of the surface-sensitive scattering modes and their results will be compared.

X-RAY REFLECTOMETRY FROM SAMPLES WITH ROUGH INTERFACES

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If x rays impinge at a grazing angle on a sample with rough interfaces, they can be reflected coherently (*i.e.* specularly) or incoherently (*i.e.* diffusely), and also transmitted coherently or incoherently. The transmitted x rays can be absorbed and excite x-ray fluorescence radiation, which can be measured in an angle-dependent x-ray fluorescence (AD-XRF) experiment.

We will show that the interface composition and roughness (both in perpendicular and lateral directions) can be obtained from x-ray reflectometry and AD-XRF experiments. X-ray standing waves, set up in layered materials, can provide the interface roughness profile and the chemical composition as a function of depth in diffuse reflection and AD-XRF measurements, respectively.

The theory of the influence of interface roughness will be discussed and experiments yielding detailed information on the interfaces will be presented.

SURFACE MELTING OF ICE REVEALED BY GLANCING ANGLE X-RAY SCATTERING

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We present glancing angle x-ray scattering experiments at (00.1), and (11.0) surface of ice I_h single crystals. The temperature dependence of the evanescent Bragg scattering upon heating reveals a quasiliquid surface layer well below the melting point on each investigated ice surface. At (10.0) and (11.0) surfaces, thermal faceting is observed, which is briefly discussed. The "oxygen-forbidden" (00.4) Bragg profiles which give direct access to hydrogen long-range order have been investigated in the bulk and close to the surface. Although in the bulk the (00.4) width is resolution limited, we discovered prior to surface melting a strong disorder of the hydrogen bonds within a mesoscopic surface sheet.

X-RAY DETERMINATION OF SURFACE ROUGHNESS OF POLISHED SILICON WAFERS

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Highly flat and smooth silicon surfaces are required in device fabrication of down-scaled microelectronics. While classical profilometers using optical and mechanical probes map micromorphologies of surfaces with super- and sub-micron lateral resolutions, scanning tunneling microscopes image topographs of a small area with atomic resolutions. X-ray scattering methods can evaluate average surface roughness with nanometers lateral resolutions, filling the gap between the techniques in current use. The X-ray methods are unique in their capability to investigate buried interfaces nondestructively. The present study applies grazing-angle X-ray reflectivity (GAXR) and X-ray scatterings along the surface normal (XSASN) to the determination of the roughness of model silicon surfaces prepared using mechanochemical polish techniques. Surfaces of four different roughness levels (2~20 Å rms) were prepared on (100) silicon wafers and examined on a triple-axis X-ray reflectometer and a triple-crystal diffractometer at a synchrotron radiation source. The same surfaces were also investigated using a state-of-art Talystep profilometer (by Jean Bennett at China Lake, California) and a WYKO optical interferometer (by M. Nakano at Shin-Etsu Handotai Co.). The Talystep used a conical stylus with a 0.7 μm tip radius, while the WYKO imaged interferograms on a CCD device with a 1×1 μm pixel size. X-ray data were analyzed by including Debye-Waller-type factors in Parratt's formulae for GAXR from multilayered structures and Cowley's formulae for XSASN (or crystal-truncation-rod) intensities to evaluate rms roughness (σ) [1]. The GAXR data allowed separate evaluations of σ's at the air-SiO_2 and oxide-silicon interfaces. The σ values calculated from X-ray measurements correlate well with roughness measured directly by mechanical and optical profilometers, but a systematic discrepancy is found between the σ values for the oxide-silicon interfaces determined from the GAXR and XSASN data. It will be discussed if this concerns the total and crystalline components of roughness at the interface or the different spatial wavelengths sensible with the two X-ray techniques.

We thank T. Abe for the sample provision.

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NONSPECULAR X-RAY SCATTERING OF INTERFACIAL ROUGHNESS IN SINGLE- AND MULTILAYERS

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A short overview is given on the different nonspecular x-ray scattering methods that have been applied to deduce information on the height-height self- and cross-correlation functions of one or several rough interfaces [1-3]. It is shown that the scaling behaviour of the height fluctuations, as predicted by the theory of kinetical roughening [1-3], can only be tested if the data is sampled over a sufficiently large Q range. A comparison between different scattering geometries is made, i.e. the so-called *rocking, detector, offset, and out-of-plane scan*, respectively. Furthermore, special forms of the height-height self-correlation function and the corresponding structure factors according to the distorted wave Born approximation [7] are discussed. For a single rough interface the x-ray scattering results are compared with measurements by atomic force microscopy. Finally, a new method to measure the degree of conformality in multilayers as a function of parallel length scale is proposed [8].

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New dynamical effects in X–ray standing waves methods

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The peculiarities of the secondary radiations yields accompanying the X–ray diffraction have been considered. The well known in the dynamical theory the Borrmann effect and the Pendelozung effect reveal itself in abig variety of different qualitative forms depending on the type of the secondary radiation, the diffraction geometry and the coherence conditions /1/. Owing to these peculiarities, the X–ray standing waves method can be applied essentially wider than it is utilized up to now.

Another very pronounced effect exists at the grazing asymmetric diffraction on the boundary Bragg– Laue transition when the diffraction beam moves exactly parallel to the crystal surface. This beamare formed inside the crystal on some distance from the surface and due to this fact piratically can not be observered in the standard diffraction experiments.

On the other hand, the cross–section of the beam appears to be very small and, as a consequence, the density of the energy flow in the beam appears to be very high exceeding the one in the incident beam on a few orders of magnitude /2/. This effect reveals as a drastical increase of the photoelectron yield /3/.

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HIGH-RESOLUTION TRIPLE CRYSTAL DIFFRACTOMETRY WITH 100 KEV SYNCHROTRON RADIATION

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The absorption of 100 keV synchrotron radiation in matter is weak and, as normally done with neutrons, bulk properties are studied in large samples. However, the k-space resolution obtained with a Triple Crystal Diffractometer (TCD) for high energy synchrotron radiation is about one order of magnitude better than in high resolution neutron diffraction.

Magnetic superlattice reflections have been measured in MnF_2, a classical antiferromagnet, demonstrating the potential of the technique for high resolution studies of ground state bulk antiferromagnetism. Recently the question of two length scales in the critical scattering at the 100 K phase transition in $SrTiO_3$ was studied on tne same sample on which most of the inelastic neutron scattering work was done at Brookhaven National Laboratory. The sharp component in the critical scattering from this sample is shown to be due to surface related effects.

The TCD studies on oxygen induced defects in annealed Czochralski grown Silicon are discussed in detail. Shape and number densities of the SiO_2 precipitates have been determined by Small Angle Neutron Scattering at the ILL, Grenoble, in samples containing different amounts of oxygen, annealed at 750°C and 1050 or 1200°C, respectively.The defect scattering due to stacking faults and dislocation loops has been identified in the diffuse scattering measured with the TCD and 100 keV synchrotron radiation at the identical samples, their diameters could be determined using a theory due to Larson & Schmatz (phys. stat. sol. (b) **99** (1980) 267). First in-situ measurements show the potential of the technique for studying the formation of these oxygen induced defects at annealing cycles relevant for industrial device fabrication.

At the PETRA storage ring, which serves as an accumulator for the HERA electron-proton storage ring at DESY and which is operated at electron energies between 7 and 12 GeV, an undulator beamline is currently under construction and should be available in summer 1995. Compared with the HASYLAB wiggler beamlines a gain in brightness by 4 orders of magnitude is expected at 100 keV and exciting new research opportunities will open up.

HIGH ENERGY X-RAY MICROSCOPY BASED ON BRAGG-FRESNEL CRYSTAL OPTICS: PRINCIPLES AND APPLICATIONS AT ESRF.

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Bragg-Fresnel optics (BFO) based on a single crystals has been proposed as high resolution and high efficient optics for hard x-rays at E>6keV. The combination of BFO with new generation of storage rings like ESRF open up the real possibility to develope microimaging and microdiffraction technique for hard x-rays
The performance and applications of the BFO was studied at the ESRF beamlines:

Optical properties experimentally measured are the following:

- **energy tunability**
- **sub-μm resolution**
- **high thermal resistivity and stability**
- **well defined shape of the focused beam**

The following applications have been realised:

- sub-micrometer fluorescence microprobe;
- linear and 2D microprobe for microdiffraction;
- 2D beam emittance and beam position monitors on the base of circular BFL;
- microfocus double crystal diffractometer;
- 10keV projection microscopy.

Test experiments on phase contrast imaging are very promissing for developing phase contrast microscopy and holography. Crystal BFO is of particular interest for microtopography and microtomography technique as well.

X-RAY TOPOGRAPHIC INVESTIGATION OF QUASICRYSTALS

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The quasicrystalline qualities (microstructure - defects) of both AlCuFe and AlPdMn grains are compared by considering Synchrotron White Beam X-Ray Topographs recorded at the LURE (Orsay - France).

Millemetric size grains of AlCuFe are grown in our laboratory by annealing quasicrystalline flakes in the icosahedral phase, at 860°C (just below the peritectic point) following the growth process developped at the CECM* of Vitry (France) while centimetric size grains of AlPdMn, grown by solidification, are provided by the LTPCM+ of Grenoble (France).

All the investigated grains display a **microstructure** built up with **subgrains**, the misorientation of which can be less than one **minute** and the size, bigger than one **cubic millimeter**.

In the topographs of these subgrains it is possible to see contrasts looking like dislocations networks, **dislocation loops** and **dislocation bands**. Dislocations loop like contrasts are seen in subgrains investigated in as-grown crystals while dislocation band contrasts appear only after mechanical polishing.

All these contrasts are presently under investigation by carriyng out systematic x-ray topographic studies (including monochromatic studies).

*CECM Centre d'Etudes de Chimie Métallurgique
+LTPCM Laboratoire de Thermodynamique et de Physico-Cchimie Metallurgiques

OBSERVATION OF DEFECTS IN CRYSTAL SURFACE LAYERS BY GID X-RAY TOPOGRAPHY

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The rising interest to grazing incidence variants of X-ray diffraction topography is connected with the prospects of application of this traditional technique for investigating the real structure of thin surface crystal layers [1-3].

In this work the possibilities of observation of single defects in real materials by plane wave grazing incidence diffraction (GID) topography were investigated both experimentally and theoretically. The experiments were performed at the beamlines ROEMO1 and CEMO of HASYLAB, using double-crystal Ge / asymmetric Si monochromators. The tunability of synchrotron radiation enabled to avoid usual limitations on applicability of GID geometry and use simple coplanar diffraction schemes.

The contrast formation on in-surface screw dislocations in lithium niobate and threading dislocations in bulk gallium arsenide and epitaxial layers on GaAs and Si substrates is discussed. The influence of pure refraction effect is shown separately on model silicon crystals with etched trenches of the depth from 25 nm to 800 nm. The results of computer simulation of dislocation images in surface layers of single crystals are presented.

It is shown, that a change of diffraction conditions from usual asymmetric to extreme GID below the critical angle of total external reflection, achieved by a slight wavelength variation, does not change qualitatively the images of near surface defects, though the distortions, produced by the defects in the underlying layers, become invisible at grazing incidence, due to both depth resolution of the method and inevitable loss of lattice parameter resolution. This allows, however, to use GID topography for characterization of films on substrates with a low perfection.

Thus, the GID topography proves to be a useful tool for investigation of thin subsurface crystal layers at the depths from tens to hundreds of nanometers and can provide information, not obtainable by usual methods.

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SIMULATION OF WHITE BEAM SYNCHROTRON TOPOGRAPHS

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White beam topographs may be simulated using the principle of reciprocity and an accurate numerical algorithm (Carvalho & Epelboin a, b 1993). We have applied these principles to the simulation of stroboscopic images of quartz resonators. We present here the result of the preliminary investigation where a new program has been written suitable for parallel and array computers. One of the main advantages of this new generation of program is that the data describing the strain in the crystal are computed separately then used in the simulation program. Thus it is very easy to change the strain model without having to change the simulation program. The disadvantage of such a technique is the need of large disc space: the derivatives of the strain must be sampled in each incidence plane using a step small enough to correctly describe the deformation. The program needs a large memory, together to store the strain and data and to be efficient in using the vector or parallel computers. It may be as large as 300 Mbytes!

Preliminary calculations on a simple strain model, already used in the simulation of section topographs (Zheng 1989) have allowed to establish the parameters for the calculation. The convergence of the numerical algorithm requires small steps in the integration of the Takagi-Taupin equations, as low as 0.01 micron along the edges of the Borrmann fan to correctly follow the phase shift of the wavefields inside the Borrman fan. The derivatives of the strain must be sampled according to the model of deformation. For the models describing the deformation of piezoelectric devices a mesh of 2x5 microns seem dense enough.

Profiles in one incidence plane have been computed and are in agreement with the experiment. First tests have been made on scalar machines, array computers (Cray) and parallel machines (TMC CM5). Other parallel machines must be investigated since the speed-up has been very unsatisfactory on this last machine. In any case the simulation of white beam topographs is a long calculation. For an image of 512x512 pixels it requires 300 hours on an IBM RS6000/560 and is estimated to 10 hours on a Cray C90.

More sophisticated models for the deformation (Yong Kong 1992) will now be studied.

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Twinning, Phase Transitions and Crystal Growth Studies from 100 to 800 K

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Morphological measurements have been precisely determined as a function of temperature for different inorganic crystals with a computer–controlled traveling Laue x–ray camera. The in–situ measurements, available over a temperature range from 100 to 800 K, provide unique insights into the kinetics of phase transitions as well as the influence of crystal surface to volume ratio on these phenomena. Premonitory phase transition behavior and the effect of temperature on existant twins are illustrated. It is hoped that such information will be of significance for understanding phase transition behavior of materials and petrological genesis of minerals.

STUDY OF PIEZOELECTRIC MATERIALS AND PIEZOELECTRIC DEVICES BY X-RAY TOPOGRAPHY

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The piezoelectric materials are obtained by two main growth methods. The first one is the hydrothermal growth method used for quartz, berlinite and phosphate gallium and the second one is a pulling method, the Czochralsky growth method, which is used, for instance, for lithium tantalate, lithium niobate and langasite. The main devices which we have study, are resonators with different cuts (AT, ST, Y for instance) and filters.

The assessment of these materials perfection by X-ray topography will be presented as some results concerning the acoustic modes and the interactions between the defects and the acoustic vibrations in the cases of bulk acoustic waves (B.A.W) and surface acoustic waves (S.A.W). The different materials have been studied by reflection or transmission topography conventionnal or stroboscopic using the white radiation delivered by the DCI synchrotron at LURE in France and a Laue setting.

To use the stroboscopic experimental set-up the resonator must be designed to have a vibration resonant at a frequency equal to n times that of the X ray synchrotron radiation which is 3.169280 MHz at DCI.

The white beam of the synchrotron radiation and the use of a Laue setting permit to record simultaneously different topographs (each spot of Laue is a topograph) with different diffraction vectors. With this technic the spatial structure of the vibration modes can be analysed and, in particular, the different stationary components of the modes can be seen separately. The stroboscopic topography, as for it, is the only technic which permits to evidence the progressive components of the acoustic modes when they exist.

Different results concerning the interactions of the acoustic waves with dislocations, in particular in a ST quartz slice (SAW) working at 9.9MHz, will be reported.

INVESTIGATION OF STRUCTURAL PROPERTIES OF InAs/AlSb SUPERLATTICES BY X-RAY METHODS

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The combination of InAs and AlSb in heterostructures offers interesting physical properties such as a large conduction band offset between InAs and AlSb, high electron sheet concentrations and high electron mobilities in InAs [1]. In the present work short-period $(InAs)_6/(AlSb)_6$ superlattices with AlAs-like, InSb-like, or alternating interfaces are studied by X-ray diffractometry, -reflectivity and grazing incidence diffraction. The superlattices are grown on GaAs substrates with a 1 µm AlSb buffer layer. Triple crystal measurements reveal that the inhomogeneous deformation of the relaxed buffer layer leads to a peak broadening corresponding to coherent scattering domains with a size of 150 nm. Double crystal rocking curves show an additional broadening and shift of the superlattice peaks, when the AlAs-like interface is grown on top of the AlSb layer. The average relaxation of the superlattices is measured using asymmetric reflections. Linear dislocation densities up to 10^6 cm^{-1} between the buffer layer and the superlattice are determined from these measurements. Rocking curve simulations show that the asymmetric peak broadening may be attributed to a further relaxation of the superlattice, which is inhomogeneous with depth. The diffusion of As into the AlSb layers leads to an additional peak shift. For the simulations we assume that every interface consists of four atomic layers of a mixed crystal InSb/AlAs with a composition of 0.5, i.e. the bond lengths show some relaxation over this distance.

Reflectivity measurements reveal quite large values for the interface roughness up to 1nm. These roughness values are probably a measure for the widths of the interface layers, since the method can only measure the differences in chemical composition. Measurements of the diffuse scattering show that this roughness is highly correlated.

The best structural quality of the superlattice is achieved when InSb-like interfaces are grown on top of the AlSb layers. Then the additional relaxation is low and almost no As is present in the AlSb layers. The reported results are in agreement with optical measurements (photoluminescence, Raman scattering) and TEM investigations [2].

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VERTICAL STRESS IN LPE-Si-LAYERS ON SiO_2/Si EVALUATED BY X-RAY DOUBLE CRYSTAL TOPOGRAPHY

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Silicon on insulator has been an aim of crystal growth for a long time. The silicon layer should be perfect as well in regard of its structural as of its electronical properties. Silicon layers grown by liquid phase epitaxy (LPE) on silicon oxide have been shown [1,2] to fulfill these requirements. These layers were produced by lateral overgrowth starting from seeding windows etched into a silicon oxide layer on a silicon wafer. These layers are about triangular lamellae extending up to 480 µm [3] on both long sides from the seeding window. Their base length at the seeding window is then up to 1 mm. The lamellae are predominantly dislocation free and atomically smooth but curved with a radius of 10...15 mm.

We will present topographs of these lamellae. It is shown that a direct reflection of a silicon lamella is observed under certain diffraction conditions only. However contrast of the lamellae is observed quite generally. This contrast is due to stress exerted by the lamellae onto the substrate. At distances up to about 50...150 µm from the seeding window the contrasts indicate a relative lattice parameter increase of the substrate perpendicular to the surface of $\Delta d/d \approx 10^{-6}$. This corresponds to a vertical tensile stress of about 1 N/cm^2. By taking topographs in several reflections a considerable shear stress could be excluded.

The curvature is obviously attributed to a tensile stress. That could be proven by underetching the lamellae, i.e. removing the oxide layer between layer and substrate. After underetching the lamellae are nearly plane and the contrast of the lamellae in the substrate reflection vanishes beside some residual contrast due to refraction at the lamella edges.

We attribute the tensile stress and the corresponding curvature to an adhesive force acting onto the growing layer. This effect may be of general importance to the growth of crystalline layers on amorphous substrates, may be as well on effectively incommensurate substrates.

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PRESENT STATUS OF THE ESRF TOPOGRAPHY BEAMLINE - NEW EXPERIMENTAL RESULTS -

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The advantages of synchrotron radiation for X-ray topography and diffractometry are well known: high intensity, continuous spectrum, tunability, polarisability and time structure. However, the present setups are still limited by their flux, the non availability of high energy photons, the obtainable resolution and the small size of the admissible sample environment devices. The characteristics of the third generation synchrotron radiation source ESRF are such that the above mentioned limitations can be overcome. This opens new possibilities for these techniques. Experiments can be carried out: 1. in real time (exposure times down to about 10^{-2}s, or using stroboscopy to 10^{-9}s), 2. using short wavelengths to investigate thick and heavy crystals, 3. with heavy and large sample environment, and 4. with high sensitivity and high angular and spatial resolution.

These new possibilities are illustrated by experiments performed on the ID11 'Materials Science' and the D5 'Optical Open Bending Magnet' beamlines. They show that a) typical exposure times for white beam topography (down to about 10^{-2}s) are about 10^{-2} to 10^{-3} times shorter than at other synchrotron synchrotron radiation topographic setups, b) the diffracted beam divergence (about 2.5 10^{-5} rad) is small enough to retain a good spatial resolution when setting the film far (1 meter) from the sample, c) with short wavelengths (in the order of 0.01nm and less) is possible to carry out topographical investigations of bulky or heavy samples or to detect weak long-range deformation fields of defects, d) monochromatic beam topographs, performed on a simple setup in a "low divergent wave" or "weak beam" mode, give clues about the actual deformations around defects

Special attention is to be paid to the heat load in white beam topography even when not working with highly absorbing and thermally insulating materials. Some criteria to estimate and minimize the effect were tested.

Finally we report the present state of the ID19 'Topography and High Resolution Diffraction' insertion device beamline, and the instrumentation designed to optimize the experimental conditions. It is expected to be fully operational in Autumn 1995.

HIGH RESOLUTION X-RAY OPTICS FOR THE 3RD GENERATION SYNCHROTRON RADIATION

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Novel high-resolution x-ray optics are being developed for the use of the extremely high brilliance of the undulator x-ray beam from the third-generation synchrotron light sources. In addition to the usual monochromatization and collimation, tailoring of coherence and polarization are important.

Plane wave x-ray optics using successive asymmetric reflections will gain new roles because they can manipulate coherency of the x-ray beam, as preliminary shown by using usual bending radiation [1]. Highly collimated and spatially condensed undulator x-rays will make the asymmetric reflection much more effective, although the spatial density of x-ray photons are diluted.

Grazing incidence diffraction, which includes the extremely asymmetric diffraction as a special case, are now being widely used for surface and interface diffraction. This geometry was applied to the depth selective diffraction topography [2], which images strain field localized to surface or interface.

High collimation of undulator x-ray beam has also opened up a new possibility of energy-tunable, mili-eV resolution monochromator by a combination of flat crystals. We proposed a nested channel-cut (+n,+m,-m,-n) monochromator [3] for this purpose, and are now working for the realization of a separated four-crystal monochromator.

Polarization manipulation, especially conversion of linearly polarized x-rays to circularly polarized ones, is another subject of x-ray optics which will find wider applications in undulator x-rays. We have developed transmission Bragg and Laue type x-ray phase retarders [4]. Helicity switching up to 100 Hz was realized [5].

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X-RAY PHASE PLATES FOR SYNCHROTRON RADIATION

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Circular polarization is an essential tool for circular magnetic x-ray dichroism (CMXD) experiments, and Compton and Bragg magnetic scattering. Circularly polarized x-rays are available from synchrotron sources below or above the orbit plane from bending magnets or asymmetric wigglers sources or from the on-axis beam of helical undulators. We present here experimental results demonstrating that the transformation of the linear horizontal polarization into circular polarization using the transmitted beam through a quarter wave plate (QWP) in Bragg or Laue geometry is an alternate and attractive technique.

X-ray phase plates use the difference between refraction indices for σ and π polarizations (perpendicular and parallel to the diffraction plane respectively) at or near a Bragg diffraction as predicted by x-ray dynamical theory. This difference depends on the offset of the incident wave $\Delta\Theta$ (difference between the angle of incidence and the center of the diffraction profile) and decreases rather slowly outside the diffraction profile. The use of the transmitted beam in Laue or Bragg geometry for rather large offsets (order of several tens aresec.) can take benfit of this property. The sensitivity of the phase plate performance to the divergene of the incident beam (order of 80 aresec.) is considerably reduced. In such conditions, good diamond crystals, with a thickness of about 1 mm and then a reasonable transmission factor have been successfully used to record CMXD spectra at different rare-earths absorption edges from Pr L_{II} (6440 eV) to Tm L_{III} (8648 eV).

The efficiency of the phase plate has been measured by comparing such CMXD spectra with those obtained with the standard technique which uses the elliptically polarized beam below the orbit plane. The experiments have been performed on the dispersive absorption spectrometer (station D11) of DCI ring (LURE, Orsay, France). The polarization rate and the intensity on the sample were higher with the QWP technique.

Finally, good results have also been obtained using beryllium plate with a mosacity of the order of 80 aresec. It will be shown that the circular polarization rate thus obtained is only slightly lower than with the diamond phase plate. The good efficiency of mosaic crystals as phase plate far from the Bragg peak can be interpreted.

Optimised Reflectivities for Diamond Single Crystals

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Abstract

Theoretical optimised reflectivities for diamond perfect single crystals are determined for use in high intensity, low emittance synchrotron radiation rings such as the ESRF (Grenoble).

Bragg (normal incidence) and b) Laue (symmetric/asymmetric) cases will be discussed and the effects of changes in effective structure factor,due to polarisation/radiation damage will be considered.

The results for a) have been published in phys.stat.sol.(a) 138, 89 (1993) and 141, K83 (1994) and show that diamond not only offers the highest Bragg reflectivity relative to Si or Ge but also possesses many additional advantages (e.g. sharper FWHM, higher thermal conductivity and Debye-Waller factor) which are ideal for the new generation of high flux, low emittance SR- facilities such as the ERSF. The application of 'normal incidence' Bragg reflectivities and transmittivities for diamond outline this crystal's eventual use for the construction of a hard X-ray free electron laser (XFEL) in a fourth generation SR-facility.

The results for b) will be published later and mainly deal with the optimisation of Laue-diffracted beam intensities through thin diamond crystals now currently in operation at the 'Troika' beamline, ESRF(Grenoble). The use of diamond single crystals in this field have the very great advantage of high thermal conductivity/low thermal bump *first* SR/monochromators with consequent minimum/zero deviation and intensity loss in the diffracted beam direction.

X-RAY CRYSTAL OPTICS FOR DIAGNOSTICS AND APPLICATIONS OF LASER-PRODUCED PLASMAS

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Laser produced plasmas have temperatures of 10^6 - 10^7 K and so emit X-ray pulses in the spectral region 0.1 to 10 nm with pulse lengths 1ps - 10ns. The emitted X-ray spectrum depends on space, time and direction. It contains important information about plasma temperature and density, and the distribution of highly ionized ions.

Schemes with crystals bent in one or two dimensions are particularly suited to obtain high resolution spectra and quasi-monochromatic images of high temperature plasmas in the spectral region up to 2.6nm[1]. The sources being investigated at present are imploding laser fusion targets[2], counter-streaming plasmas[3], X-ray laser plasmas and laser-target interactions with sub-picosecond laser pulses. When the generation of X-rays is optimized, sources of incoherent or coherent X-ray pulses will become available for real time experiments and will equal or surpass current undulator or wiggler sources in brilliance.

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SYNCHROTRON X-RAY OPTICS EXPERIENCE AT THE ESRF

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A short overview of the research and development that has been carried out at the ESRF in the field of hard x-ray optics for synchrotron radiation in the past few years is presented. This includes four topics: mirrors, single crystals, multilayers and the particular question of how to solve the high heat load problem. Also focusing issues will be addressed like sagittally curved crystals, meridional focusing by multilayers and two-dimensional focusing by toroidal mirrors.

Although time constraints restrict this presentation to a more general level several highlights are mentioned like the performance of diamond at very high heat loads, the efficiency of phase plates based on Si, diamond and Be single crystals, the development of supermirror-multilayer structures and bimorph benders made of piezoelectric ceramics.

DEPTH RESOLVED ANALYSIS OF THE STRAIN PROFILE IN SEMICONDUCTOR SUPERLATTICES USING X-RAY GRAZING INCIDENCE DIFFRACTION

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During epitaxial growth of semiconductor superlattices (SL) a considerable lattice parameter mismatch must be accomodated. As long as the lateral lattice parameter difference between the layers is small (coherent growth) the total energy of distortion is taken up by the layers. In case of a larger difference partial lattice relaxation appears which depends on the total thickness of SL t_{tot} and the thickness ratio between the sublayers. A closer investigation of the real structure of multilayers requires methods which are able to evaluate these structure parameters with respect to the depth of SL. In the direction of growth the lattice mismatch is typically measured by conventional high resolution diffractiometry. Because the penetration depth of the probing X-ray beam is much larger than t_{tot} only averaged parameters can be obtained. The lateral lattice mismatch can only be given indirectly and therefore rather inaccurate.
Depth resolution is obtained by the technique of grazing incidence diffraction (GID)[1]. In this case the "information depth" t_{inf} is controlled by the angle of incidence α_i and exit α_f of the X-ray beam with respect to the sample surface. The lateral lattice parameter can be obtained in different depths below the surface. Additionally, the vertical density profile is probed measuring the intensity distribution along α_f ("rod scan") for different α_i. For $\alpha_i < \alpha_c$ (α_c - critical angle of total external reflection) t_{inf} is reduced to the top layer and/or the upper SL-period. With increasing α_i further layers contribute to the scattering signal. In addition to the "surface peak" which is visible at $\alpha_f = \alpha_i$ "SL-peaks" appear for special $\alpha_i = \alpha_{si}$. The angular separation between the α_{si} is a measure for the SL-period.
This technique has been successfully applied at the example of a lattice matched SL of $Ga_{0.5}In_{0.47}As/InP[001]$ grown by a MOCVD [2] containing an enlarged quantum well (EQW). Measuring the rod scans at the anglular position of the (220) in- plane Bragg peak we found a double peak behaviour of the recorded SL peak. The intensity ratio between both subpeaks varies changing α_i. This allows an accurate determination of the depth of the EQW below the surface and its thickness in an accuracy better than 5%. A further example demonstrates that the GID technique is able to identify buried single monolayers within perfect semiconductors.
A variation of structure parameters with depth is obtained for various strained layer $Ga_{0.8}In_{0.2}As/GaAs$ SL's grown on GaAs[001] by MBE [3]. The thickness of the individual active layers t_i is chosen smaller than the critical thickness for building of misfit dislocations. Keeping t_{tot} fixed the degree of relaxation increases for decreasing thickness of GaAs barriers t_b. Depth resolution demonstrates that the majority of misfit dislocations is localized close to substrate interface. Within the SL microdomains appear with a lattice parameter mismatch to the surrounding lattice. They are inclined up to some 1/10 deg. with respect to the surface normal. The averaged relaxation degree is nearly constant over the whole SL exept if the SL is covered by a thick top layer. Then we find in fact a variation of the in-plane lattice parameter with depth. This may be understood by a stepwise lattice deformation of the GaAs top layer near the interface to the SL.

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X–Ray triple crystal diffractrometry characterisation of defects in lattice mismatched epitaxial structures

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Over the last few years X–Ray triple crystal diffractometry has proved to be a powerful tool to analyze a diffracted intensity in two directions in the scattering plane. We report here a comprehensive X–ray diffraction study examining heterostructures (HS) with a misfit of lattice parameters in the interfaces. The objects under study were the following, $Si_{1-x}Ge_x/Si$ HS, GaSb layers strongly mismatched to GaAs substrates, YBaCuO films grown on various substrates etc. Symmetric and asymmetric Bragg,Laue and grazing incidence settings were used.

It was shown that the shape of the diffraction curves indicated the type of the defect arrangement, namely the Coloumb type or dislocations. Since the latter are known either to form regular networks or to propagate thrugh the layers, we have studied the patterns due to the both cases and the basic points of the identification each of them has been established. Triple axis setting permits an assessment of the structural quality across the layer depth. With an analyzer remaining fixed, we made a series of theta – scans and evaluated the depth of dislocation networks as well as the location of the Coulomb type defects from the shape and the position of the peaks.

Dealing with strongly mismatched HS, we considered the peak broadenings as being due to the contributions coming from three sources. One was a delation provided by randomly distributed dislocations inside the layers. Second, there was a tilt associated with grain boundaries or the dislocation arrays. Third was the finite size of coherent scattering regions. When we plot the dependence of the broadenings in two directions on the Bragg angle, we extracted and analyzed the contributions. Two of them, the strain and the tilt , were used for the identification of the dislocation types in the following way: since the dislocations Burgers veetors derive from the asymmetrical tensor of crystal lattice distorsions, they can be found from the extracted components by using various diffraction geometries.

The X–ray study of nanostructures with a misfit accomodated by dislocations was discussed.

We considered the problems associated with a simulation of diffraction curves from HS containing defects inside the layers. The ways of obtaining interplanar spacing depth profiles were shown.

The structural parameters determined from the X–ray data were in a good agreement with TEM results.

High Resolution X-Ray Diffraction from Epitaxial Multilayered Surface Gratings

the influence of the fine structure near the 000-reciprocal lattice point

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X-ray diffraction from epitaxial multilayered surface gratings has been studied experimentally by a high resolution triple crystal diffractometer and theoretically in terms of a semi-dynamical diffraction theory based on a second order distorted wave Born approximation.
Laterally periodic super-structured crystals show a two dimensional fine structure in the vicinity of their reciprocal lattice points formed by equispaced truncation rods perpendicular to the crystal surface. The diffraction pattern along a truncation rod depends on the grating shape function and the intrinsic setup of the grating period. In our case the surface gratings are formed by epitaxial and partially etched superlattices giving rise to superlattice diffraction peaks in the grating truncation rods.
Additionally the fine structure near the 000 reciprocal lattice point causes multiple beam diffraction also in the planar part of the sample below the grating. As a result the planar multilayer influences the grating truncation rods of non-zero order and dominates partially its diffraction pattern.

EXPLOITATION OF N-BEAM DYNAMICAL EFFECTS IN PSEUDO-KOSSEL DIFFRACTION PATTERNS OF NEARLY PERFECT CRYSTALS

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Divergent-beam X-ray diffraction patterns that subtend a substantial solid angle at the specimen exhibit an abundance of intersections of Kossel cones. This applies equally whichever of the three well-known methods of pattern excitation is employed; the three methods being, respectively, the original Kossel & Voges (1935) arrangement in which the specimen crystal was itself the anticathode of the X-ray tube, the method of Seemann (1930) using an X-ray tube providing a wide cone of X-rays to fall on the specimen crystal, and the present-day popular method utilising a SEM electron beam to produce a small source of characteristic X-rays within a thin film of the appropriate element in contact with the specimen. In the last method X-rays are emitted into essentially a 2π solid angle within the crystal; it is referred to as the pseudo-Kossel method.

Kossel cone intersections can be classified into **A-points, accidental,** that involve any two cones corresponding to diffraction vectors $\mathbf{g}_1$ and $\mathbf{g}_2$, with Bragg angles θ_1 and θ_2, when the angle α between $\mathbf{g}_1$ and $\mathbf{g}_2$ is such that $\alpha < \pi - \theta_1 - \theta_2$. These intersections give 3-beam diffraction conditions: $\mathbf{0}$, $\mathbf{g}_1$, $\mathbf{g}_2$. More complicated are points on the Kossel pattern where three or more lines (i.e. loci of intersection of Kossel cones by the X-ray film) have a common crossing point resulting from crystal symmetry. These points of **systematic** simultaneous reflection, **S-points,** are produced by Bragg planes that are tautozonal, provided that the common line of intersection of their corresponding reciprocal lattice planes has a segment lying within the sphere of reflection. At S-points n-beam ($n \geq 4$) simultaneous diffraction occurs. In the transmitted divergent beam patterns given by highly absorbing but perfect crystals, strongly increased anomalous transmission is observed at A-points and S-points (Borrmann 1959, Borrmann & Hartwig 1965, Hildebrandt 1966, Post, Chang & Huang 1977.) The present work describes new observations of **fine-structure,** on the scale of a few arc seconds, that can be detected within the relatively large overlap area of wavelength-dispersion-broadened intersections of Kossel lines. Besides their theoretical interest, these fine-structures superimpose 'markers' on an otherwise relatively diffuse diffraction pattern, and should be exploitable in lattice-parameter measurements.

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THE STUDY OF TRANSMISSION PHASE CONTRAST OF NON-CRYSTALLINE OBJECTS WITH A HIGH-RESOLUTION X-RAY DIFFRACTOMETRY

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In recent years a new imaging technique appeared, using the double-crystal arrangement to study low-absorbing non-crystalline objects [1-4].The object is illuminated with a beam produced by a perfect crystal monochromator. The object itself may have negligible absorption, but it introduces the phase modulation into the transmitted beam, which is visualized with help of the perfect-crystal analyzer. The wide variety of experimental applications is accumulated now (see [4]). But the image formation in this technique was not studied thoroughly yet.

Two main versions of the technique are known: the first one, [1-2], uses the analyzer crystal in Bragg (reflection) geometry, the second one, [3-4], - in Laue (transmission) geometry. Despite their apparent similarity, these two versions are rather different regarding the image formation and the contrast sensitivity. Here we analyze the image formation with Laue-type analyzer. The object of study is a polyethylene tube filled with water, containing also the air bubbles. This objects contains two types of boundaries: air-polyethylene and polyethylene-water, giving rather strong and very weak phase changes correspondingly, allowing to estimate the sensitivity of the method. In order to trace out the influence of the analyzer thickness, the wedge-shaped analyzer was used. Both transmitted and diffracted beams were registered after the analyzer. The images were taken at different deflections from exact Bragg analyzer position.

In Bragg position the object boundaries are seen black (decreased intensity) in diffracted beam and black or black-and-white in transmitted one. Moreover, the white image, if seen, is located always on the same side from the black one, corresponding to the direction of the incident beam and not depending on the sign of the phase gradient. This can not be explained on the assumption made in [1-2], that the contrast is due to the deflection of beams refracted in the object outside the Bragg reflection of the analyzer. The image in transmitted beam would be only white in this case. The explanation is given on the base of the dynamical diffraction theory - the known effect of the angular amplification of small deflections from the Bragg position creates shadow in the center of the Borrmann fan in the areas with strong phase gradient, and this effect holds for both transmitted and diffracted beams. The white image in transmitted wave is produced by deflected rays, which have essential intensity only along the incident wave direction. Depending on the sign of the phase gradient these rays belong to different wave fields in the crystal, so the brightness of the image varies also due to anomalous absorption. Other features are discussed also and sample calculations are presented.

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STRUCTURAL CHARACTERIZATION OF GaAs GROWN AT LOW TEMPERATURES (LT-GaAs) BY MOLECULAR BEAM EPITAXY

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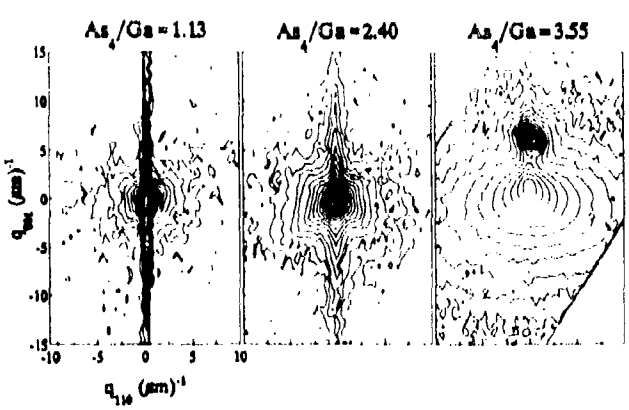
The growth of GaAs at low substrate temperatures (LT-GaAs) results in two major modifications to the otherwise "normal" crystallographic structure of GaAs: (1) the incorporation of extremely high levels of excess arsenic in the as-grown LT-GaAs layer; and (2) the nucleation and growth of As-precipitates when LT-GaAs is annealed at elevated temperatures following epitaxial growth. X-ray diffraction is ideally suited for the analysis of as-grown and annealed LT-GaAs and can yield information concerning either the "average" structure of the epitaxial layer (such as the average lattice parameter or the degree of strain accommodation with the substrate) or statistically distributed distortions in the LT-GaAs lattice. We have used high resolution x-ray diffraction methods to determine that despite the incorporation of more than 1% excess arsenic, layers of as-grown LT-GaAs can exhibit crystallographic perfection superior to that of the substrates on which they are deposited. The structural quality of the LT-GaAs was found to depend strongly on the ratio of the As_4 and gallium fluxes used in the MBE growth. High resolution triple crystal x-ray diffraction scans (shown below) demonstrate that at low flux ratios, the LT-GaAs was virtually indistinguishable from the substrate. At higher flux ratios, we observe (1) an increase in the magnitude of diffuse scattering as well as an anisotropy in the scattering, and (2) a degredation in the crystal truncation rod. Further increases in the As_4/Ga ratio resulted in copious diffuse scattering. We have developed a model of point defect aggregates that at least partiallyl accounts for the observed diffuse scattering. Differences have also been observed in the diffuse scattering from *n*- and *p*-doped LT-GaAs. Analysis of annealed LT-GaAs layers using high resolution diffraction methods has shown the development of long-range strain fields within the epitaxial layer that arise from distortions of the GaAs lattice due to the growth of the arsenic precipitates. We have used the excess diffuse scattering that surrounds the Bragg reflection to quantitatively determine the sizes and concentrations of arsenic precipitates in annealed LT-GaAs. The average size of the arsenic precipitates determined from these diffuse scattering analyses are in resonable agreement with precipitate size measurements made with electron microscopy. Using these methods of x-ray analysis, the formation of arsenic precipitates in LT-GaAs can be understood as a process that is similar to the much-studied oxygen precipitation in Czochralski silicon.



(left) 004 triple crystal x-ray diffraction scans from LT-GaAs grown by MBE with different As_4/Ga flux ratios.

CHARACTERIZATION OF III/V-HETEROSTRUCTURES GROWN BY SELECTIVE AREA EPITAXY USING DOUBLE-CRYSTAL X-RAY DIFFRACTOMETRY WITH HIGH LATERAL RESOLUTION

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There is a particular interest in characterizing III/V-heterostructures grown on patterned surfaces by laterally resolved X-ray diffraction. For this purpose, a new convergent-beam double-crystal X-ray diffractometer has been developed. The X-ray beam is focused onto the sample by means of a cylindrically curved mirror as a sharp line focus. A position sensitive proportional counter is used for the parallel recording of rocking curves along the line focus. The instrument allows the parallel recording of several hundred rocking curves, each representing an area of 20 x 40 μm^2 of the sample.

Using selective area epitaxy (SAE) epitaxial growth occurs locally in unmasked areas defined by dielectric material deposited on a semiconductor substrate. We investigated SAE-samples that were grown by two different techniques: metal-organic vapour phase epitaxy (MOVPE) and metal-organic molecular beam epitaxy (MOMBE). The hetero-structures consist of ternary and quaternary layers based on the Ga-In-As-P material system deposited on InP-substrate. The aim of our experiments was to determine the layer mismatch approaching the growth/non-growth boundary. Therefore rocking curves were recorded across the mask edges. The rocking curve measurements show variations of the stochiometry at the dielectric/semiconductor boundary of MOVPE grown ternary layers. These variations indicate an increasing In-content at the mask edge. This effect does not depend on the crystallographic orientation of the mask edge. The stochiometric inhomogenities are less pronounced if the growth rate of the layers has been reduced. The position resolved rocking curve measurements describe the distorted regions of the grown layers near the mask edge

Our results have been confirmed by position resolved photoluminescence measurements. Investigations of different MOMBE grown samples did not show any variation of the lattice mismatch close to the growth/non-growth interface. From this it may be concluded that the MOMBE SAE enables undisturbed growth up to the mask edge.

DETERMINATION OF THREADING DISLOCATION DENSITY IN HETERO-EPITAXIAL LAYERS BY DIFFUSE X-RAY SCATTERING

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We report on a novel approach for the determination of dislocation densities based on the analysis of the two dimensional (2D) intensity distribution of diffuse x-ray scattering measured in the diffraction plane by triple axis x-ray reciprocal space mapping. It is based on Holy's formalism for the simulation of experimental isointensity contours using either the mosaic block model (MBS: piled-up dislocations) or the model based on random elastic strain fields (RED: randomly oriented dislocations).[1] The corresponding two sets of parameters which quantitatively describe the defect structure in the epitaxial layers are used for calculating the threading dislocation density. For the MBS model, the mosaicity parameters (mean block size and misorientation) were used according to Gay et al.[2] However, for the RED model the new ansatz is a caculation based on an energy balance. The simulation of the 2D intensity contours yields the random strain field from which the energy density stored in the epilayer due to the presence of defects was calculated. On the other hand the self energy of threading dislocations is calculated assuming 60° or screw type dislocations and taking the crystal ansiotropy into account. The threading dislocation density is obtained by dividing the energy density by this self energy. The procedure is iterative, the calculation of the dislocation self energy requires an initial value of the outer cut off radius, i.e. of the spacing of the threading dislocations.

This method was applied for the analysis of five SiGe layers (B2) grown on compositionally graded SiGe alloy buffer layers (B1) deposited on (001) Si substrates. The buffers B1 are graded up to nominally 30% Ge in steps with different grading rates (10, 20 and 40%Ge/μm) yielding threading dislocation densities in the buffer layers B2 (x_{Ge}=30%, 1μm thick) from $5\times10^5 cm^{-2}$ to $1\times10^7 cm^{-2}$ as determined from EBIC data.[3] (004) and (224) reciprocal space maps of the substrate and the buffers were analyzed. Our analysis of the diffuse x-ray scattering reproduces the increase of the dislocation densities in B2 with higher Ge grading rates quite well, and moreover yields upper limits for the threading dislocation densities which are about a factor of 10 higher than those deduced from the EBIC data. However, if dislocation interaction is taken into account in the analysis, this factor reduces to about three.

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X-RAY DIFFRACTION FROM MESOSCOPIC SYSTEMS: THIN FILMS ON "ROUGH" SURFACES

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The morphology of thin films on top of laterally structured surfaces (here Si surface gratings: spacing 1 μm, height 130Å) was studied by x-ray diffraction in the region of total external reflection [1].
Polystyrene films with thicknesses of 200Å to 700Å were deposited on the samples. The reflected intensity yields the averaged density profile of the system, whereas the q_z-scans along the positions of the diffraction orders of the grating are sensitive to the in-plane structures of the films. A theory developed by Joanny, Andelmann, and Robbins [2] which is able to describe the experimentally observed behaviour of cyclohexan films on rough (but mesoscopically flat) Si substrates [3] predicts a rather rapid decay of the amplitude of the periodic structure of the topmost surface. Already for film thicknesses of more than 200Å and above a nearly flat surface is expected. In contrast to the theoretical predictions of the experiment the "roughness" is visible even for polymer film thicknesses of 700Å. This observation was additionally confirmed by AFM measurements.
Semiconductor films (here Ge/Si-multilayer) on top of these gratings show a quite different behaviour. Here the x-ray measurements yield that the films (10 films Ge/Si, each film 50Å thick) are totally conformal with the surface grating which means that they follow the underlying lateral structure without decaying of the grating height. The conformity is not only observed for the mesoscopic structures (shape of the films) but also for the microscopic roughnesses (interface roughnesses).

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REAL-TIME SYNCHROTON RADIATION TOPOGRAPHY OF THE PARA-TO-FERROELECTRIC PHASE TRANSITION OF AMMONIUM SULPHATE

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Ammonium sulphate, $(NH_4)_2SO_4$, transforms at -49°C from the orthorhombic paraelectric room-temperature phase (space group Pnam) into the orthorhombic ferroelectric phase ($Pna2_1$). Besides other interesting features the transition is accompanied by a considerable change of the lattice parameters with a dilatation by 1.07 % along the a-axis and contractions of 0.23 % and 0.31 % along the b- and c-axes, respectively (Hoshino et al., 1958).This leads to a hyperbolic representation surface of the transition strain tensor with directions of zero dilatation on the asymptote cone of the hyperboloid. The phase boundary will adopt such directions of zero dilatation (low-energy boundaries) and develop a zigzag shape with segments parallel to the two symmetrically equivalent minimum-strain orientations (Hussinger & Unruh, 1977).

The phase transition of ammonium sulphate and the special features of its phase boundary have been studied by LAi topography and by white synchroton radiation topography (Bhat et al.) Here we rero .al synchroton radiation study (SRS Daresbury) applying a real-time X-ray imaging This allows the continuous topographic display of the phase transition on a TV monitor a cord on a video tap. The high-resolution imaging system and the low-temperature cine mber are described. A sequence of pictures taken from TV records shows the following features of the transition:

- The movement of the phase boundary through the crystal and the development of its zigzag shape.
- The formation of cracks at the acute apices of the zigzag phase boundary.
- Boundary segments of minimum-strain orientation do not induce damages on moving through the crystal, whereas segments of other orientations imply high strain and leave behind a trace of defects.

The support of this study by the SERC is gratefully acknowledged.

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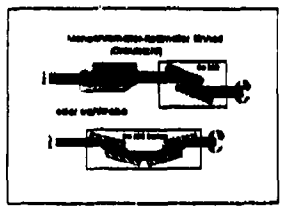

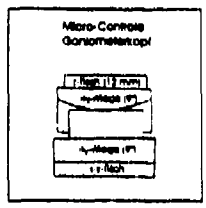

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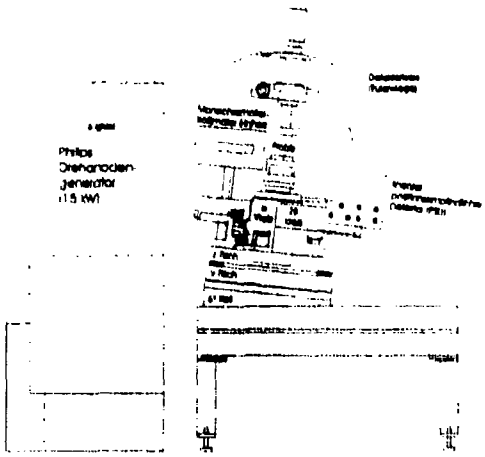

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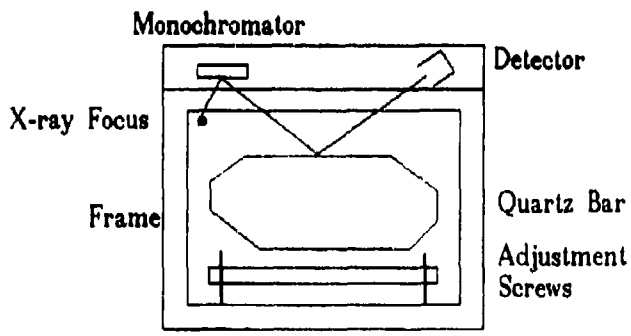

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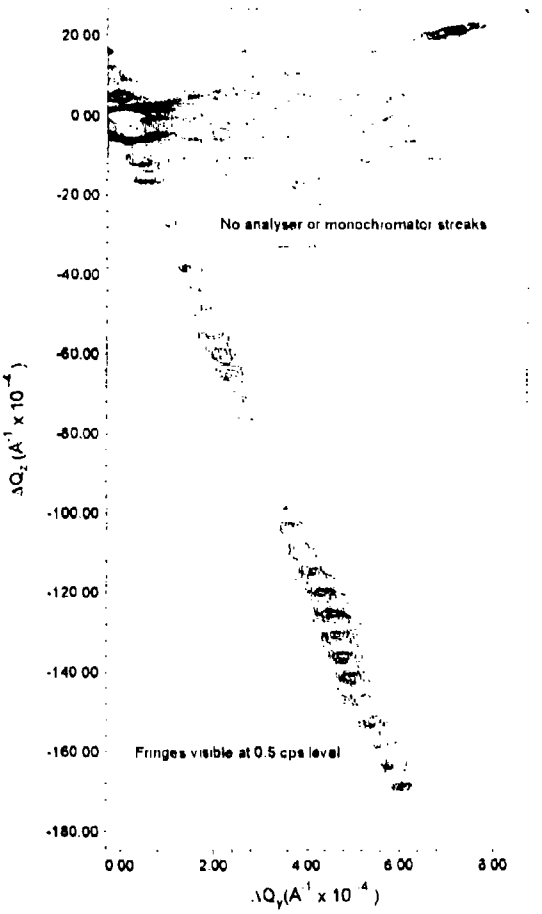

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DIFFUSE SCATTERING OF X-RAYS IN MULTILAYER STRUCTURES

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Diffuse scattering of x-rays in multilayer structures results from the roughnesses of interfaces, impurities near boundaries due to the interdiffusion, and bulk non-uniformities due to the microcrystalline structure of the different layers. We report here the results of the experimental and theoretical studies on the angular spectra of scattered radiation.

The angular spectrum of scattered radiation exhibits the interference maxima, when the wave vector of the scattered wave obeys the condition $\vec{k} = \vec{k_0} + \vec{H_n}$, where $\vec{k_0}$ is a wave vector of the incident wave, and $\vec{H_n} = \vec{e_z}\ 2\pi n /d$ is a reciprocal vector of the multilayer structure. The intensity In of the n-th maximum is proportional to the $\left|\chi(\vec{s_l})\right|^2$ [1], where $\vec{s_l} = \vec{k_l} - \vec{k_{0l}}$ is a longitudinal projection of the scattering vector on the interface plane, and $\chi(\vec{s})$ is a Fourier- transform of the dielectric permitivity $\chi(\vec{r})$. Beyond the angles of the Bragg diffraction for the incident wave the interference maxima due to the interface scattering, therefore we can determine the correlation length for the interface roughnesses by measuring the intensity of the interference maxima as a function of the incidence angle.

The measurements of the scattered wave intensity, when the incident wave passes through the Bragg maxima, enable us to determine the ratio of bulk and interface scattering intensities. This is due to the appearance of the x-ray standing wave which results in the appearance of the interference maxima for bulk scattering. The x-ray standing wave enables us also to separate the scattering due to the boundaries between the A-B layers and B-A layers. We study the effect of the forbidden reflection release.

We use the $Cu\ K\alpha$ x-ray emission line, and multilayer mirrors with the different pairs of materials (W/Sb, Fe/Sc, Mo/Si, Mo/B_4C). The periods of the structures are in the range from 20 up to 200 A. The mirrors with the silicon and glass substrates are used in our experiments.

The intensities of the diffuse maxima (n = 1..4) are measured in the broad range of the incidence angles. The behavior of the intencity of the diffuse maximum with n = 2 is thoroughly studied when the angle of incidence varies in the region of the first Bragg reflection maximum. The dependences of the angular spectra of scattering on the ratio of the layer thicknesses b/a (where $d = a + b$) are investigated. The effect of the release of the forbidden reflection in the scattered radiation is demonstrated for the mirrors with $b/a = 0.5$. The experimental and theoretical spectra are compared.The estimations of the statistical parameters of the non-uniformities for the different mirrors are given.

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Investigation of Polarization Characteristics of Reflected Waves under Grazing Geometry X-ray Diffraction.

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The unique possibilities of grazing geometry X-ray diffraction for investigation of crystal surface structure and thin films stimulate the detail analysis of this phenomenon in the recent time both theoretically and experimentally [1-4]. Despite the great interest to this diffraction scheme the polarization phenomena have not yet been investigated.

In this paper the transformation of the X-ray polarization under grazing geometry diffraction in the two-wave approximation of the dynamical diffraction theory is analyzed. The dependence of polarization of specularly reflected and diffracted in vacuum waves on corresponding parameters of incident wave with an arbitrary polarization is found. As critical angles of reflection for eigen σ- and π-polarizations do not coincide the scattering of the wave with polarization different from the eigen one leads to changing of polarization state of the secondary waves. In the case when polarization plane of the incident linearly polarized wave is oriented under the angle of 45^0 to the crystal surface the degree of circular polarization of the reflected waves can reach the value ~ 0.5.

Polarization characteristics of reflected waves strongly depend upon the surface structure of crystals. The influence of the amorphous layer on the degree of circular polarization of specular and reflected diffracted waves is investigated. It is shown that at a certain thickness of amorphous layer it is possible to transform incident linearly polarized radiation into the circular one with the degree of circular polarization close to unity. The case when the amorphous film is anisotropic (for example, magnetic) is also considered. The effect of excitation of the waves with orthogonal polarization under 90^0-diffraction of π-polarized beam in the crystal with magnetic amorphous layer is analyzed. Grazing geometry conditions lead to the essential increase of the angle of Faraday rotation with respect to normal incidence. Analysis shows that even far from the absorption lines when the difference between the refractive indices for the waves with right-hand and left-hand polarization is small ($n_+ - n_- \approx 10^{-7}$ and less) the effect can be used for investigation of magnetic films with the thickness 10-100 nm.

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DETERMINATION OF YBaCuO THIN LAYER STRUCTURAL PARAMETERS BY USING HIGH RESOLUTION X-RAY DIFFRACTOMETRY.

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In this paper, an X-ray diffractometry in a double and triple axis settings was applied to hTS $YBa_2Cu_3O_{7-x}$ layers with the thicknesses from 200 to 600 nm grown on MgO, $SrTiO_3$ and sapphire substrates by a magnetron sputtering. Only monophase and C-oriented films were studied. The series of 00l symmetrical Bragg reflections was measured and analyzed in two directions in the scattering plane.

The diffraction of the layers has been treated like that of the mosaic crystals. As such, the full width of half maxima (FWHM) obtained in the double axis arrangement are considered to be consisted of the contributions which are as follows: the finite size of coherent scattering regions or grains, the tilt of the grains and the dilation of the crystal lattice caused by dislocations. By utilizing triple axis theta- and theta-2 theta scans we plotted the dependencies of FWHM values on the Bragg angle. It was shown that the theta-scan curve broadenings were primarily due to the deviation of C axis from its predominant orientation. The strain contribution was found to be substantially smaller than the tilt one. A detailed analysis of the low reflecting orders allowed us to evaluate the average grain size.

When dealing with YBaCuO layers by using an X-ray diffractometry, one should be aware of the fact that it is the oxygen nonstoichiometry which defines the lattice C parameter as well as the scattering factor of the unit cell. We discussed a possible determination of X value from the layer C parameter measurements.

From the integral reflectivities of 00l Bragg reflections we calculated F^2t values, where F is the unit cell scattering factor and t is the layer thickness. Then F(X) dependences were plotted according to the data presented in /1/, and F as well as t values were determined.

The structural parameters of the layers were studied in association with those of the substrates. The defects in the latter ones, namely, the dislocations, their arrangements and damage layers, were revealed by using an X-ray diffraction topography.

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X-RAY DIFFRACTION STUDY OF THE LATTICE STRAIN RELAXATION IN MOVPE GaAs/Ge HETEROSTRUCTURES

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GaAs/Ge heterostructures can be conveniently used as basic materials for fabricating GaAlAs/GaAs/Ge high conversion efficiency solar cells. One advantage of the GaAs/Ge system is that the mismatches in both the lattice parameter $\Delta a/a$ and the thermal expansion coefficient $\Delta\alpha/\alpha$ are small ($\Delta a/a$=-0.07%, $\Delta\alpha/\alpha$=15%). However, for solar cell applications the GaAs layer is usually a few mm thick, thus well above the critical thickness for the lattice strain relaxation; the critical thickness calculated on the basis of the elastic equilibrium theory is indeed 0.28 μm. Therefore, the mechanism of the strain relaxation through the formation of extended defects and the correlation between defects and growth procedures are worthy of experimental investigations.

GaAs/Ge heterostructures have been grown by Metal-Organic Vapour Phase Epitaxy with different V/III flow ratio (1.3 to 13.3); the layer thickness was always much larger than the critical threshold for the elastic strain relaxation. The structural properties of the specimens have been investigated by both X-ray topography and high resolution X-ray diffractometry. In the first case a conventional Lang camera in Bragg reflection conditions (Cu $K\alpha_1$ radiation, 115 asymmetric reflection) was used. In the second the investigation was done by a Philips diffractometer equipped with a two-crystal four-reflection monochromator (Ge, 220); the Cu $K\alpha_1$ radiation and a set of 2 symmetric 004 and 4 asymmetric 335 reflections were used for measuring the lattice parameter both parallel and perpendicular to the interface.

It has been found that at high V/III ratios the layers are affected by a large concentration of stacking faults, whereas misfit dislocations are completely absent; the fault density decreases by decreasing the V/III value. A small, but significant strain relaxation has also been observed, thus demonstrating the effectiveness of the partial dislocations bordering the faults in relaxing the strain. At lower V/III ratios the dominant defects are the misfit dislocations to which the strain relaxation can be attributed.

INFLUENCE OF Te DOPING ON LATTICE PROPERTIES OF AlGaAs EPITAXIAL LAYERS

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*** High Pressure Research Center Unipress, Polish Academy of Sciences, ul.Sokolowska 29/37, 01-142 Warsaw, Poland*

Tellurium is one of the most important n-dopants in III-V semiconducting compounds. In this work we present the results of measurements leading to a full understanding of an influence of Te atoms on the lattice state of AlGaAs layers. The examinations were performed with a high resolution diffractometer at a wide temperature range (77-770 K). The following problems were considered.

a) how Te doping influences the real structure of the layers,
b) how Te doping influences the thermal expansion of the layers,
c) what kind of lattice relaxation occurs when DX centers are transferred into their metastable state.

In order to solve these problems we have examined samples with different Al content and Te concentration (of different minima of the conduction band and of different DX level position). Special attention was paid to the separation of the effects caused by ionized donors and by free electrons. The X-ray diffraction measurements (rocking curves and lattice constants) were supported by the electrical examinations (Hall effect).

Acknowledgements

This work is supported by the grant No 2 P3 02 08305 of the Polish Committee for Scientific Research

Investigation of the 3-dimensional Structure of Pb-Stearate Multilayers by GID and X-ray and neutron specular reflectivity

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Universität Potsdam, Institut für Festkörperphysik; [b] Universität Mainz, Inst. f. Phys. Chemie; [c] Universität München, Sektion Physik, Lehrstuhl Peisl

Multilayers of fatty acid salts prepared by the Langmuir-Blodgett (LB) technique on solid support build structures that are well ordered in growth direction.

The X-ray specular reflectivity measures the electron density gradient normal to the surface. Using this technique it is possible to determine the thickness of a double layer, the total thickness of the film and the roughness of the CH_3 - Pb-ion interface. The interface between the hydrocarbon chains is invisible. In contrast to this neutron scattering is sensitive to the gradient of the scattering length. The gradient can localized at the interface between the carbon chains, when some chains are deuterated. Respective experiments we demonstrated performed at a home X-ray reflectivity equipment and at the beamline TOREMA II at the reactor in Geesthacht, Germany.

The analysis of the lateral molecular arrangement within Langmuir-Blodgett-multilayers requires the detection of a sufficient number of in-plane Bragg peaks. They can be measured using the grazing incidence X-ray diffraction technique (GID).

We have investigated Pb-stearate multilayers ($1 < N < 19$ monolayers) prepared on Si-support by means of angular dispersive GID at the W1 wiggler beamline at HASYLAB at DESY in Hamburg, Germany ($\lambda = 1.44$ Å, $E = 8.61$ keV, $\Delta E \quad 1$eV) . Choosing the angle of incidence smaller than the critical angle of total external reflection α_c the information depth is reduced to the thickness of the LB film. The diffracted intensity of the Bragg peaks was measured in-plane (2θ-scans) and along the surface normal (rod-scans), using a position sensitive detector. Sixteen different Bragg peaks were found within $0 < 2\theta < 70$, independent of N (for $N > 1$), which corresponds to a nonhexagonal in-plane arrangement of molecules. The rod-scans were measured at six peak positions and between them ($0 < \alpha_i < 14$). We found that the in-plane arrangement of the molecules corresponds approximately with the 3D structure of bulk material, when $N > 2$. The vertical correlation of the lateral order is restricted to 1-2 double layer.

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THIN SILICON CRYSTALS AS POSSIBLE MONOCHROMATORS FOR THE ESRF TOPOGRAPHY BEAMLINE

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One of the crucial points in the monochromatization of the X-rays at a third generation synchrotron radiation facility is the handling of the heat load on the first monochromator crystal (or first optical element). This makes it necessary to apply complicated cooling systems, to use weakly absorbing materials, and/or sophisticated geometrical arrangements. For the ID 19 ESRF topography beamline (about 250 W for a closed gap, 100 mA electron beam current, and only a 1.5 mm Be window as filter), which uses a wide beam (height 14 mm and width 40 mm) in an energy range from about 6 keV up to more than 100 keV, the requirements with respect to the homogeneity are very demanding. The best material in the case of the topography beamline remains silicon. One possibility to reduce the power of the incoming radiation absorbed by the monochromator crystal, is to make it thin [1]. Depending on the foreseen energy range, a compromise for the crystal thickness must be found between a minimum of absorption and a maximum of reflectivity. Furthermore, the crystal plate must be highly perfect, i.e without lattice defects like dislocations, and the bending of the reflecting lattice planes must be less than the Darwin width for the considered energy (about 5 arc seconds or less). For other applications (e.g. monochromators for coronary angiography at HASYLAB) such solutions were already tested [2], but not for the requirements for topography.

We investigated several silicon wafers with a thin central window (membrane) of variable geometry, produced by the Fraunhofer-Institut für Siliziumtechnologie in Berlin. The thicknesses of the membranes were between 1µm and 5µm. To characterize the structural perfection of these samples we used several methods: 1) optical interferometry (measurement of the surface profile and in some cases the roughness with a Wyko 6000) 2) white beam X-ray topography (detection of crystal defects and crystal deformations using the topography stations at the synchrotrons LURE and ESRF) 3) triple crystal X-ray diffractometry (detection of net plane bending, depth profile of deformation, ...) with instruments at the Grenoble University (MRD from Philips) and the Optics beamline of the ESRF. It was possible to characterize the main parameters of the thin silicon membranes which determine their suitability to be used as Laue (transmission) case monochromators for synchrotron radiation at higher energies.

The results of the first tests are very promising. In some cases the structural properties already approach the level we need (bending of the membrane, its thickness fluctuations)

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FERRIMAGNETIC DOMAINS AND THE VERWEY TRANSITION IN MAGNETITE

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Magnetite, Fe_3O_4, exists as a mineral and is the oldest magnet. It is a ferrimagnet and is cubic above its Curie temperature of 858 K. At room temperature, its easy magnetization directions are <111>. It undergoes at about 120 K the Verwey transition to a phase where the easy magnetization directions, referred to the high-temperature cubic axes, are <100>, and where complex distortions and atomic displacements lead to magnetoelectric behavior which is still not fully understood.

Good single-crystal samples, with thickness in the range 0.2 to 0.5 mm, of magnetite grown by the Bridgman method were investigated by white-beam synchrotron radiation topography, using a temporary setup on bending magnet line D5 at ESRF, operating at 6 GeV. The beam was filtered with 0.5 mm of aluminum and 0.6 mm of iron. The topographs were recorded on Kodak Industrex R film set perpendicular to the incident beam at a distance of 16 cm from the specimen. Alternatively, a Sofretec image intensifier CCD camera with a P45 phosphor was positioned to follow the image in real time on a selected Bragg-diffracted spot. The observations could be performed also below the Verwey transition, using a closed-cycle Displex type helium refrigerator, and a magnetic field of about 0.2 T could be applied.

At room temperature, simple domains are observed in a $(01\bar{1})$ plate, containing four easy magnetization directions in the surface. The domains are observed through the 71° and 109° walls, with orientation close to (011) or (100). Strong variations in their contrast is encountered, depending on the wavelength used for the reflection and on the corresponding structure factor, as well of course as on the direction of the scattering vector relative to the walls. Spectacular anomalous contrast was observed when a very weak reflection, 711, was enhanced through simultaneous reflection (Umweganregung) of 400 and 311, at a wavelength of 0.23 Å: the domains then showed up as *area* contrast, although their difference in distortion, due to magnetostriction, is very small ($\lambda_{111} \approx 8.10^{-5}$). The same sample only has two easy directions, ± [100], parallel to its surface below the Verwey transition, resulting in a complicated domain structure with closure domains, both without magnetic field and with a field applied in an arbitrary direction during a preliminary experiment. Short movies of both the domain wall movement under a field and the phase transition could be recorded on a microcomputer.

These results will be shown and discussed. So will the results of subsequent investigations involving the application of the field in a well controlled direction, and other samples, aimed also at the search for variations in distortion in the magnetically saturated state.

The authors are happy to thank Mr. S. Todo for preparing a single crystal and Prof. T. Doy for polishing two of the samples used.

X-RAY PHASE FRESNEL ZONE PLATES AT GRAZING INCIDENCE

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Recent developments in X ray optics afford the production of a new focusing element: a Bragg Fresnel lens (BFL), which is a perfect crystal with a Fresnel zone profile of the crystal surface [1]. A BFL has a high spatial resolution of about an outermost zone size and a high focusing efficiency (up to 40%). It has also a narrow spectral bandwidth of about $\Delta\lambda/\lambda \approx 10^{-3} \div 10^{-4}$. Such a bandwidth is due to a high spectral sensitivity of Bragg reflection. However, quite a number of research and applied tasks do not require such high monochromatization.

To broaden the spectral bandwidth we proposed [2] to abandon Bragg reflection and use the total external reflection. Moreover, the total external reflection has some useful properties: the reflection coefficients of wavelengths are closely to unity, the penetration depth of waves inside the scattering medium is about tens of angstroms and the use of the reflection layout allows the production of focusing elements as phase ones. The new type of X ray focusing elements proposed are phase Fresnel zone plates at grazing incidence and they were called grazing incidence phase Fresnel zone plates (GIPFZPs).

In this work the configuration and the optimal zone height of GIPFZPs have been defined for 1-D X ray (sagittal and meridional setups) and 2-D X ray focusing. The spectral and spatial resolutions, the intensity at the focal spot have been defined for these GIPFZPs as well. Linear and elliptical GIPFZPs have been fabricated. An experimental testing of these focusing elements has been carried out using an X ray generator with a rotating anode ($\lambda = 1.54$ Å). A high contrast of the source images show the potential possibilities to use the GIPFZPs for X ray microprobes.

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THE USE OF THE X-RAY TOPOGRAPHY IN THE INVESTIGATION OF TWINNING IN $LaGaO_3$ SINGLE CRYSTALS

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A detailed study of a real structure the $LaGaO_3$ single crystals, grown by the method of crucibles zone melting with radiation heating, has been attempted by the methods of X-ray topography and diffractometry in this work in a wide thermal range (20°-300°C), including a probable phase transition interval at 150°C [1].

It has been found out that at 20°C these crystals have an orthorhombic unit cell with the parameters: a=5.520(2)Å, b=5.490(2)Å, c= 7.770(1)Å and twinning in {110}/<1$\bar{1}$0> and {112}/<1$\bar{1}$0> systems simultaneously. The regions of cross-twinning have been discovered in these systems.

The first-order phase transition from orthorhombic to rhombohedral lattice has been observed at 139.5°C. The temperature hysteresis of the transition was about 0.5°C. The crystallographic parameters of the rhombohedral cell being a=3.889Å, a=89.50° (150°C). The samples in high temperature phase have also a developed twin structure. The inherited transformation of twin structures occurs during the phase transition, at which the cross twinning in {100}/<001> of a rhombohedral lattice transforms into the twinning in {112}/<1$\bar{1}$0> of an orthorhombic phase. Long-term cycling through the point of the phase transition retains only one {112}/<1$\bar{1}$0> twinning system.

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THE DYNAMICAL DIFFRACTION EFFECTS IN THE X-RAY TOPOGRAPHY OF THE HIGH TEMPERATURE SUPERCONDUCTORS

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In this investigation we observe the X-ray anomalous transmission effect in high temperature superconductors (Nd_2CuO_4, $(LaSr)_2CuO_4$, $EuBa_2Cu_3O_{7-x}$ and $YBa_2Cu_3O_{7-x}$) of different thickness (15-100 μm) [1].

With the of X-ray topography it is shown that the $EuBa_2Cu_3O_{7-x}$ crystals grown in alundum containers are noncentrosymmetrical. The image of the crystal reverses its contrast on opposite after rotation by 180°. The space group of such crystal is P2mm. They have a polar axis along [100] and are divided into polar and antiphase domains. The polarity and ferroelectricity of the HTSC are discussed in literature [2-4].

For the $EuBa_2Cu_3O_{7-x}$ crystals we obtained the dependence of the Borrmann effect from the length wave. At the λ=0.95A the image of the crystal changes its contrast.

The X-ray topographic image of the single crystals $YBa_2Cu_3O_{7-x}$ and $EuBa_2Cu_3O_{7-x}$ consists of the domains with boundaries along (110) ($YBa_2Cu_3O_{7-x}$) and (100) ($EuBa_2Cu_3O_{7-x}$). Variable concentrations during the growth or the antiphase twinning are likely to be the reason for this structure.

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THE INFLUENCE OF COHERENCE LENGTH OF PSEUDO PLANE X-RAY BEAM TO THE CONTRAST OF DEFECT IMAGES.

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The multicrystal plane wave topography is effective in detection of micro defects and other sources of small deformations in perfect monocrystals. Due to incident plane wave the computer experiment is possible for some types of defects. However the results of computer simulations often do not coincide with experiment. Of course, imperfections of the defect model may be the reason, however it is obvious that pseudo plane wave parameters are also important. Various theories exist (e.g., [1,2]) taking a plane wave divergence into a consideration and we've made some experiments to test their results. We had examined the images of different defects in various plane wave schemes, (n, -n) and (n, -n, n), with different wave parameters in MoK_{α} radiation and Si FZ-monocrystals.

Experimental results showed the strict dependence of image character on the pseudo plane wave divergence. The primary wave divergence increase leads to the defect image expansion. This result coincides with the theoretical conclusions made in [2]. The primary wave divergence decrease results in X-ray optics scheme resolution rise so as the images of large dislocation loops become more informative. The method sensitivity to small deformations becomes better. However when the tie point moves to the rocking curve tail the images of small power defects disappear because the size of kinematic scattering area correlates with the grain size of the photo emulsion and the interference is suppressed.

The images of small power defects also disappeared in any tie points in experiments with the triple crystal spectrometer where the wave divergence was near 0,08". This result was not predicted in theory and can be qualitatively explained by the presence of additional peaks in the rocking curve of perfect monocrystal [3].

When the crystal under test is deviated from the exact Bragg position there really exist some layer in the crystal center, in which the interference phenomena are disappeared (it is not in conflict with [1]). The shorter is the coherence length the thicker is this layer and it appears faster when tie point moves to the rocking curve tail. The direct qualitative observation of said statements with the help of inclined dislocation model is also supposed. So the angular position of the crystal under test close to the exact Bragg one is the most informative in the plane wave scheme due to fulfillment of the conditions of interference contrast.

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High resolution X-ray diffraction study of porous silicon

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Porous silicon (PS), obtained by an HF electrochemical etching, was first investigated for Silicon On Insulator technology applications. More recently, the discovery of the optical properties (photoluminescence and electroluminescence) rose a considerable interest for this material. In addition to its optical properties, PS exhibits interesting physical properties. For example PS remains a nearly perfect single crystal even for large porosity (above 80%).

Using high resolution X-ray diffraction, we have investigated porous silicon samples fabricated under different conditions. For p^+ type on (001) wafer orientation, the PS lattice parameter is slightly increased compared with the substrate one. The investigation of an asymetric reflection (4 -2 -2) shows that the interface between PS and the substrate is coherent. At the bottom of the Bragg peaks, reciprocal space maps show an anisotropic diffuse scattering which is due to the nanometric size of the silicon nodules. For p type (with a smaller pore size) the lattice expansion is larger than for p^+ type. We have measured lattice parameter variations induced by various effects: anodic oxidation, presence inside the pores network of a liquid or an alkane vapor...

MOSSBAUER FILTRATION OF SYNCHROTRON RADIATION AT ISOTOPE INTERFACE AT GRAZING INCIDENCE

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Mossbauer filtration of Synchrotron Radiation (SR) [1] due to the resonant Mossbauer scattering at an isotope interface (a plane interface between different isotopes of the same chemical element) under conditions of a total internal reflection is theoretically investigated. The mentioned scheme of Mossbauer filtration of SR allows one to suppress the background connected with the nonresonant SR scattering because of absence of reflection at the isotope interface if there is no resonant scattering. Calculations of the reflection coefficient at the $^{56}Fe/^{57}Fe$ isotope interface versus the SR energy and the incidence angle are performed. It is shown that due to the sharp resonant dependence of the total reflection critical angle for an isotope interface on the SR energy the choice of the incidence angle and of the divergence of the incident SR beam allows one to change in a great extent an energy line width of the filtrated SR. Because in any real experiment other interfaces except of the isotope one are inevitably present and generate a nonresonant background special geometries of these interfaces which allow to suppress the background are examined. The main idea of the suppressing of the background by a choice of the interfaces geometry reduces to the choice of such geometries in which the isotope interface is nonparallel to the another ones. This allows to separate the scattering direction at the isotope interface from the background scattering directions from the other interfaces with a simultaneous background intensity reduction because the nonisotope interfaces occure to be far away from the total reflection condition. A vacuum – non Mossbauer isotope interface of a saw-like profile combined with a planar isotope interface is examined in details. Numerical calculations illustrating advantages of the proposed scheme of SR Mossbauer filtration in comparison with the others are carried out for a $^{56}Fe/^{57}Fe$ isotope interface.

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CRYSTALLINE PERFECTION OF THE PEROVSKITE [Nd, Sr](Ta, Al)O_3 STUDIED BY OPTICAL AND X-RAY METHODS

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Cubic solid-solution perovskites with the composition $[Nd_{0.4}\ Sr_{0.6}](Ta_{0.3}Al_{0.7})O_3$ to be used as lattice-matched substrates for epitaxial growth of high-T_c superconductor layers were grown by the Czochralski process. Specimen slices were prepared from seed, middle, and tail sections of the crystal boule. They have been characterized by photoelastic studies, chemical etching, double-crystal topography and rocking-curve measurements as well as by triple-axis diffractometry (mapping around the reciprocal lattice point).

The halfwidths of rocking curves measured on various points of the slices vary appreciably (22" to 140"). In the region of the half radius of a slice, the lowest values seem to be realized. All the curves show broadened shoulders. In areas with growth disturbances, the rocking curves are broader and splitted, frequently.

Striking features of the defect structure are variations of the chemical composition on a large-range scale and in growth striations, revealed by chemical etching as well as by X-ray topography. The amplitude of the relative lattice-parameter variations over the striations amounts (1 to 2) $\times 10^{-4}$. The whole difference of relative lattice parameters in an area of about 3 mm^2 was found to be in the order of magnitude 8×10^{-4}, which corresponds to composition variations of about 1 at%. From the reciprocal-lattice mapping, it follows that the lattice-parameter changes are correlated with orientation changes, probably due to non-cubic lattice distortions that also mainly contribute to the observed photoelastic contrast.

γ/γ' MISMATCH STUDIES IN A SINGLE-CRYSTALLINE Ni-BASE SUPERALLOY BY RECIPROCAL-LATTICE MAPPING

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Ni-base superalloys are important as materials with high-temperature strength. They consist of two phases, the fcc γ phase and the γ' phase having an Cu_3Au superstructure. The lattice mismatch $\delta = 2(a_\gamma - a_{\gamma'})/(a_\gamma + a_{\gamma'})$ between both phases effects the creep behaviour. Therefore, it is necessary to determine this mismatch and further structural parameters in order to understand the strength and creep properties of these materials.

Because of the small mismatch and of the broad orientation distribution, the full two-dimensional distribution in reciprocal space has to be studied. Reflections with high diffraction angles ($\Theta \approx 70°$ and 74°) were used to obtain large lattice-parameter changes. The lattice-parameter distributions were integrated over all orientations. Three samples of slightly different composition, each before and after creep deformation were investigated.

The intensity maxima of the two phases are only partially separated so that the mismatch as well as their volume fractions had to be determined by profile fitting. Symmetric curves (Gaussian, Lorentzian) do not give good reliability in some cases so that asymmetric curves were taken into account. Superstructure reflections of the γ' phase were measured additionally to indicate the shape of single-phase reflection profiles.

The mismatch values amount from -5×10^{-4} to -6×10^{-4} for undeformed and from -8×10^{-4} to -12×10^{-4} for deformed samples. This difference seems to be large considering the applied stress of about 70 MPa. The volume fractions of the γ' phase vary between 50% and 70%, which is also an unexpected high value.

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THE EXTINCTION LENGTH OF THE X-RAYS DIFFRACTED BY THE ONE-DIMENSIONAL MODULATED STRUCTURES

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The X-ray and the extreme ultraviolet radiation diffraction by the periodicone-dimensional modulated multilayer interference structures (MIS) is considered. In the case of the perfect semi-infinite MIS there exists a minimum number n of the layers, effectively reflecting in the diffraction region of the incidence angles. This number is defined by the condition $n \gg L / d$, where L is the extinction length and d is the space period of the MIS. If the angle of incidence $\hat{U}$ is far from the diffraction angular region, then depending on $\hat{U}$ for the real absorbing MIS there exists a finite maximum number $n(\hat{U})$ of the structure layers, which are interacting with the incident wave field. This number is defined by the condition $n(\hat{U}) \gg L(\hat{U}) / d$, where $L(\hat{U})$ is the depth of penetration. The parameter $n(\hat{U})$ may be changed by the corresponding selection of the materials, as well as of the relations between the layer thicknesses in the one period of the MIS (e.g. see [1, 2]). By this the reflection coefficient $R \to 1$, if the conditions $N \gg n$ and $n << n(\hat{U})$ are simultaneously fulfilled, where N is the real MIS layers' whole quantity. In particular the MIS reflectivity is increasing while the L is decreasing. In view of this in the paper it is investigated the L of the one-dimensional modulated MIS. It is shown in particular, that even in the case of the perfect transparent MIS with the periodic one-dimensional modulated polarizability (the case with cosine-like polarizability is considered in [3]) the angle of incidence, which is corresponding to the extinction length, does not coincide with the middle of the angular region of the unstable solutions.

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X-RAY SPECULAR STANDING WAVE

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In the case of Laue geometry of diffraction and the backscattering from the monocrystals and the high quality multilayer interference structures (MIS) the diffraction angles of incidence corresponds to the region of the total external reflection. One of the two waves reflected from the structure surface is well-known as a specular (mirror) reflected wave, and the other one is the plane wave reflected from the structure in the opposite direction of the incident wave (specular backscattered wave, see [1, 2]). In consequence of the interference of these two specular waves is forming a specular standing wave (SSW) directly over the structure surface with the same space period as for the reflecting family of the net planes. The dispositions of the SSW crests and nodal points along the structure surface are depending on angle of incidence and the Bragg angle. One may determine the dispositions and the length of the chemical bond of the foreign atoms, adsorbed on the pure structure surface, by measuring the secondary radiation and shifting SSW along the structure surface. Moreover, one may obtain the impurity atoms two-dimensional disposition pattern by combining of this method with the ordinary one in which the distances are measured in the case of the Bragg geometry of diffraction from the other family of the net planes of the same structure.

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STRAIN IN THICK EPITAXIAL LAYERS

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X-ray diffraction measurements between room temperature and 400 ^{0}C with a precision of $\Delta d/d \approx 10^{-4}$ (Bond method) exhibit some details in the variation of strain in $\sim$1 μm thick epitaxial layers of GaAs, InP, CdTe, EuS or SrS on Si or GaAs substrates. These layers grow coherently at a lattice mismatch $f \lesssim 0.001$ between layer and substrate and incoherently at larger f values. The slope $\Delta\epsilon''/\Delta T \approx \alpha_s - \alpha_o$ of the strain ϵ'' parallel to the substrate is negative or positive because of the different thermal expansion coefficients α_s and α_o of the substrate and thin film materials. The lattice parameters of the cubic layers, which are deposited at high temperatures T_1, deviate from the lattice parameters of the single crystals at this temperature (fig. 1). Therefore a strain $-10^{-3} \lesssim \epsilon^0 \lesssim 10^{-3}$ remains on cooling to point A, which can be much larger than the strain $-10^{-5} \lesssim \epsilon^0 \lesssim 10^{-5}$ in large single crystals. The ϵ^0 values of all planar GaAs and EuS samples at point A are increased by one or two annealing processes at 160 – 400 ^{0}C to point B or C. Negative ϵ^0 values were observed for GaAs, InP and SrS, positive ϵ^0 values for EuS. Approximately constant ratios $-(\epsilon^\perp - \epsilon^0)/(\epsilon'' - \epsilon^0)$ are obtained, if the ϵ^0 values at A, B or C are considered as reference values instead of the $\epsilon^0 = 0$ values of the single crystals. These ratios can deviate for different layers of the same material, e.g. between 0.48 in 425 nm EuS and 1.27 in 500 nm EuS layers. These values are also different from the values 0.75 (EuS), 0.90 (GaAs), and 1.13 (InP), which are obtained from elastic constants. A large variation of the ϵ^0 and T_1 values at points A, B and C changes the physical properties or destroys the epitaxial layers. Small variations are achieved

a) for systems with a strong bonding between layer and substrate as e.g. InP/GaAs.
b) for GaAs, which was stabilized at the steps of a vicinal Si (001) plane.
c) for GaAs layers with amorphous buffer layers. The temperature of point B of these layers corresponds with the temperature of deposition of the buffer layer. The layers are unstrained at point B and do not vary on further annealing.

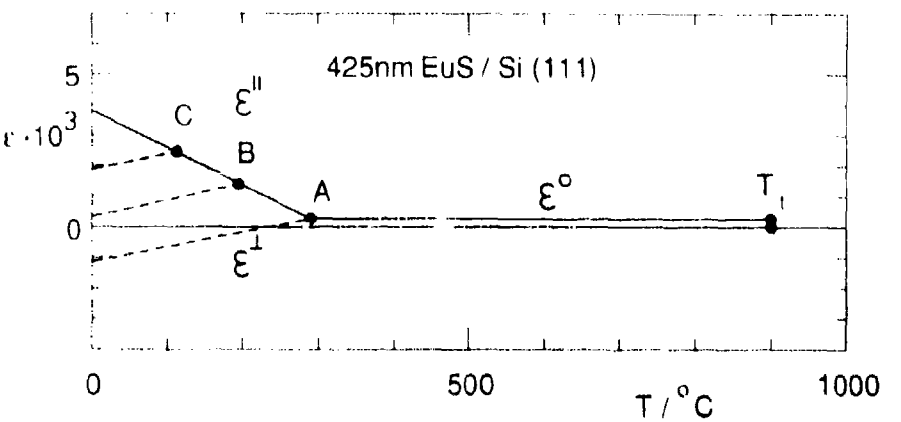

Fig. 1: Variation of strain in a EuS layer after deposition at T_1 (A) and after annealing at 260 and 280 ^{0}C (B, C).

ABSTRACTS
HIGH TEMPERATURE TRIPLE CRYSTAL X-RAY GONIOMETER

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A triple crystal goniometer with high temperature furnace has been designed. This goniometer is capable to provide high resolution x ray measurement at temperatures up to 850°C. The goniometer ensure a sufficient angular and an excellent temperature stability. It is possible to use inert gas ambient at atmospheric pressure.

A 3μm ZnTe layer deposited on 500μm GaAs substrate was studied using this equipment. Thermal expansion coefficient of the substrate has been measured using the triple crystal arrangement in the temperature range up to 350°C. With this method the Bragg angle is measured directly (relative value with respect to room temperature position) and thus any angular instabilities of the heated sample are eliminated.

Combining both triple and double crystal measurement we have determined thermal expansion coefficient and elastic constants c_{11}, c_{12} of the layer. Stress in thin film was measured from the radius of curvature of the substrate. Elastic deformation was determined from the substrate and layer peak separation in both symmetrical and grazing exit asymmetrical diffractions.

In addition the elevated temperature reciprocal space maps of the layer have been measured in symmetrical and two asymmetrical diffractions.

The apparatus can be used for studying of inelastic effects in thin films which occur during high temperature annealing. The results of the latest experiments will be shown too.

X-RAY STANDING WAVE STUDY OF CdTe/MnTe/CdTe (100) HETEROINTERFACE

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Some structural characteristics of 0.7 and 2 monolayers of MnTe buried in a CdTe(100) matrix were studied by means of the x-ray standing wave (XSW) method. The XSW results were interpreted with the dynamical theory of x-ray diffraction. According to this theory, standing waves occur in the crystal as a result of the interference process between incident and diffracted wave fields. The standing wave pattern has the periodicity of the reflecting planes. By rocking the crystal within the angular range of the Darwin curve, the standing wave pattern shifts by half of a period. Therefore, a layer of buried atoms will have variation of fluorescence depending on there positions and the known nodal-antinodal positions.

The XSW experiments were performed on the set-up which is installed on the beam line D25B of the LURE (Orsay France). Two sets of experiments were chosen ; one with CdTe004 symmetrical reflection (Ge311 monochromator) and the other with CdTe113 inclined reflection (Si220 monochromator). We find the MnTe layers to be pseudomorphically matched to the CdTe host crystal. The position of the MnTe atoms, the crystalline quality and some type of defects have been accurately determined and will be reported.

THE INFLUENCE OF X-RAY DIFFUSE SCATTERING ON THE SECONDARY PROCESSES YIELD

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One of the most perspective methods of investigation of the disturbed surface layer structure is X-ray high resolution diffraction with simultaneous registration of the angular dependence of the secondary processes (SP) yield (photoemission, fluorescence). The intensity of SP is proportional to the intensity of total X-ray field. The theory of SP is usually based on the assumption of completely coherent scattering. The presence of structure defects is taken into account by introducing the static Debye-Waller factor f only. However it is well-known that defects give rise to a diffuse component of the scattering intensity. The diffraction reflection curve and SF yield are the sums of coherent and diffuse components.

In the present report the statistical dynamic theory of diffraction is used to discuss the influence of defects in a disturbed crystal surface layer on the angular distributions of coherent and incoherent (diffuse) scattering, and on the angular dependence of SP yield. The following effects are taken into account: 1) the diffuse absorption of coherent fields, 2) the SP, which are generated by the diffuse scattered quanta, 3) the multiple incoherent rescattering of diffuse scattered intensity (secondary extinction). Calculations are made for a disturbed layer with a constant lattice deformation and randomly stress deformation of statistically distributed microdefects with variation within a wide limits of static factor f (from 1 to 0), Kato correlation length, lattice deformation, crystal and disturbed layer thicknesses.

QUANTITATIVE CHARACTERIZATION OF CRYSTALS FOR X-RAY SPECTROSCOPY

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In the fields of x-ray spectroscopy and x-ray plasma diagnostics, characterization of flat and bent crystals employed as an optical element in Bragg optics is needed to accurately interpret measured spectrum. In particular, in the relative soft x-ray range, e.g. λ=0.2-0.8 nm, which is important for spectroscopic analysis in plasma diagnostics, fusion studies, and dispersive EXAFS, the integrated reflectivity data of both flat and bent crystals have hitherto not been quantitatively evaluated.

Based upon the x-ray kinematic and dynamic diffraction theories, the integrated reflectivities of numerous crystals, namely, Quartz, PET, ADP, InSb, RAP, KAP, and OHM have been calculated, and analyzed by taking into account the effects of the x-ray wavelength, the bending radius, and the crystal perfection.

The integrated reflectivity measurements of those crystals have been performed at BESSY KMC beam line with a triple-axis diffractometer [1] and at our laboratory with a dispersive achromatic configuration [2], and with a hybrid monochromator which provides both high energy and angular resolutions [3]. The agreement between the calculations and the experimental results is satisfactory. In particular, the influences of the crystal bending perfection on bent crystals, which are revealed by using x-ray topographic technique, are demonstrated, and are in accordance with corresponding theoretical calculations.

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3-DIMENSIONAL DISTRIBUTION OF THE CRYSTAL ORIENTATION IN AN IRON-3% SILICON ALLOY SINGLE CRYSTAL SHEET OBSERVED BY X-RAY ORIENTATION TOPOGRAPHY

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<Aim> In observing an iron-3%Silicon alloy single crystal sheet by X-ray scattering topography (microbeam scanning type apparatus)(1), we recognized some differences of the surface reflection image from the transmitted one. It implies a structure distribution along the depth even in a sheet as thin as 0.2 mm. Thus we have made 3-dimensional distribution of the crystal orientation inside a single crystal plate cut from an Fe-Si alloy sheet having GOSS structure. The secondary recrystallization mechanism(2) is also discussed.

<Methods> We have obtained orientation distribution maps on the (110) surface by the X-ray orientation topography(3) after step-wise thinning from the thickness 0.2 mm. The X-ray orientation topography is consisted of a one-axis orientation topography and a reconstruction-mode orientation topography(4).

<Results> 1)The orientation varies in an angular range of about 22 minutes in every layer. 2)Almost half of the orientations are seen within 2 minutes in every layer. 3)The orientation changes rapidly near the grain boundary in every layer. 4)The orientation changes rapidly in the vicinity of the both surfaces. 5)A few small subgrains are found at limited locations inside the sheet. 6)From these 3-dimensional observation we could estimate a growth mechanism of the secondary recrystallized GOSS structure.

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THE CONTRAST OF GROWTH LAYERS IN Ge-Si CRYSTALS BY X-RAY TOPOGRAPHY

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ABSTRACT

In the present paper the growth layers of Ge-Si crystals are investigated by X-ray topography, Lang method, using MoK_α (0,707 Å) and AgK_α (0,559 Å) on the (111), (220), (422) reflection planes.The crystals have been obtained by Czochralsky method in [111] growth direction having a variable Si-impurities concentration - 0,25 to 4 atomic percentage -. The rotation velocity has been 30 rot/min to 100 rot/min.Using the results of the dynamical X-ray diffraction theory, the studied crystals exhibits black and white bands which are crystalline regions with opposite curvatures.The width of the growth layers is depending of the rotation velocity; their contrast has a dynamical character.There are discussed, also, the contributions to the total lattice deformation given by bending distortion and the lattice spacing change, according the Kato's theory.A low-order reflection planes leads to a better resolution and the width of the growth layers is larger when a longer wavelength is used. It is pointed out, also, the manner in which the contrast of the growth layers obtained by X-ray topography arises from a different scattered intensity by the distorted lattice, being mainly determined by the amount of migration of the tie points on the dispersion surface branch.

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X-RAY RECIPROCAL SPACE MAPPING OF GaAs/AlAs QUANTUM WIRES AND QUANTUM DOTS

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We have investigated periodic arrays of 150 nm-wide [110] oriented GaAs/AlAs quantum wires and dots, fabricated by e-beam lithography and $SiCl_4/O_2$ reactive ion etching, by means of reciprocal space mapping using triple axis x-ray diffractometry (TAD). From the x-ray data the lateral periodicity of wires and dots, and the etch depths are extracted as well as the occurence of a small strain gradient. Information about the shape (height, width, inclination of the side walls ...) and shape fluctuations of the wires was obtained by comparison of the maps with two dimensional kinematical simulations. The reciprocal space maps reveal that after the fabrication process the in-plane lattice constant of the patterned multilayer sample does not change, whereas the lattice constant along the growth direction slightly increases for the wires and even more so for the dots.

The GaAs/AlAs quantum wires and dots were realized by nanostructuring a 30 period AlAs-GaAs multi quantum well (MQW) grown on a 1 µm GaAs buffer. The 8 nm thick GaAs wells are separated by 12.8 nm AlAs barriers resulting in a total thickness of 625 nm. Wire satellites accompanying not only the SL_0 peak but also the first order MQW-peak SL_1 (up to the second order) are observed. The wire period determined from the spacing of the satellites along the lateral q_x direction is 303 nm. In an equivalent measurement for the dot array satellites up to the third order have been observed. D_i denotes their respective order. In the measurements the sample was oriented either with [110] direction or with [100] perpendicular to the diffraction plane. Consequently the lateral periodicity was changed from 300 nm to $300\cdot\sqrt{2}$ nm. In the latter case this increased period manifests itself in a decrease of the dot satellite spacing by a factor of $1/\sqrt{2}$. The dot period determined from the distance of the dot satellites in q_x direction is 310 nm from the first case and 302 nm from the latter.

An anticipated elastic relaxation after fabrication would result in a decrease of the average lattice constant in growth direction $a_\perp^{MQW}$. However, from the measurements follows, that on the contrary, the nanostructured MQW is even more strained in the growth direction (q_z direction). The shift of the zero order wire satellite with respect to the SL_0 peak of the unstructured MQW corresponds to an enlargement of $a_\perp^{MQW}$ from 5.663 Å in the reference sample to 5.669 Å in the wires, and to 5.673 Å in the dots. This effect is most probably due to the oxidation of the AlAs layers during or after the etching

DETECTION PROJECT FOR THE 'TOPOGRAPHY AND HIGH RESOLUTION DIFFRACTION' BEAMLINE OF THE E.S.R.F.

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ESRF will re-open the fields of 1) high resolution diffraction and topography and 2) in situ topographic studies of phenomena evolving with time whose kinetics are too fast to be investigated with presently existing storage rings. For both fields detectors are a crucial part of the instrument. A substantial effort is being devoted to this topic in order to make the best possible use of the ESRF source.

Ideally we would need simultaneously a high dynamic range, a large quantum efficiency, a spatial resolution [illegible] µm and the possibility of recording over 40 ms with a dead time [illegible] 10 ms. This detector does not exist. Developments of prototypes are very expensive and we chose the best compromise by using a combination of commercially developed devices. The planned detector ensemble (fig. 1) includes CCD cameras equipped with a visible light optics and scintillators, films, and energy sensitive proportional counters. This ensemble is coupled with specific computer driven image acquisition and treatments. For stationary phenomena noise is decreased by averaging over several images and subtracting the flat field. A sliding memory buffer is to be used for the investigation of rapidly evolving effects, like first order phase transitions. The filtering of the images is foreseen as a post acquisition treatment.

Several kinds of detectors are presently being used:

1) films (Kodak type R or High Resolution) with ~1 µm resolution. Typical exposure times at the ESRF for type R films are in the range 10^{-3}–1 s for large area topographs and ~20 times more for a section topograph. For time resolved work developments are foreseen in mechanical cinema methods in which a stack of films are rapidly exposed in series.

2) CCD video cameras (CCIR 625) looking at a scintillation screen through a microscope type objective lens. For thin high quality scintillating screens this approach offers micron level resolution and the possibility of variable magnification. Screen thickness (5-200 µm) can also be varied to exchange resolution (~30 µm) for quantum efficiency but the latter will always be poor [illegible] difficult to produce. Cooled CCD systems are already available with ~$10^3 \times 10^3$ pixels, and single frame dynamic range ~10^4 for slow readout (~2 sec). It should be noted that fast readout CCD [illegible] by data storage considerations.

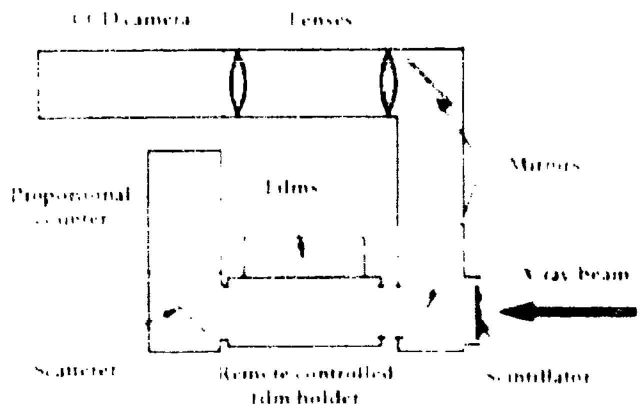

Fig. 1

QUASI-MONOCHROMATIC IMAGING OF LASER-PRODUCED PLASMAS

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Spatially resolved monochromatic two-dimensional images of laser-created plasmas have been recorded by using toroidally bent crystals. Quartz, silicon and [illegible] high diffraction efficiencies and to maintain crystal perfection during bending. Ray-tracing codes were developed to calculate the [illegible] for given experimental setups using flat, cylindrically, spherically and [illegible] bent crystals. [illegible] spatial resolution better than [illegible] to minimize the geometrical aberrations [1]. Comparison of intensities in images of different x-ray lines allows one to evaluate plasma parameters such as electron temperature and spatial distribution of special ions in certain excited states. Monochromatic images of fusion pellets driven with the 10 kJ Gekko XII glass laser system at Osaka University are presented in this paper [2]. The electron temperature is determined by seeding the deuterium fuel with argon and evaluating the intensity ratio of the two monochromatic images of the Lyβ (Ar^{+17} $1s\,^2S_{1/2}$ - $3p\,^2P_{3/2}$) and Heβ (Ar^{+16} $1s^2\,^1S_0$ - $1s3p\,^1P_1$) lines.

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CHARACTERISATION OF HOMOEPITAXIAL GROWTH OF SILICON AFTER DC-HYDROGEN CLEANING WITH X-RAY ROCKING CURVES

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Cleaning of silicon wafers are important processes in microelectronic device fabrication. The control of these processes are critical and characterisation of the cleaned surfaces are necessary. Here, a DC Hydrogen plasma cleaning procedure is investigated [1,2]. High resolution X-ray rocking curve (HRXRD) measurements were performed and compared with secondary ion mass spectrometry (SIMS) depth profiles of the substrate-epilayer interface, by cross section transmission electron microscopy (XTEM) and Rutherford backscattering (RBS) channelling. HRXRD measurements give information about the lattice strain in function of the depth of semiconducting materials and thin films. The HRXRD technique is a non destructive method with the highest strain sensitivity of any known technique. It could be shown, that the sensitivity of the HRXRD method is good enough to detect defects of importance for microelectronics. It was also possible to detect the influence of very small contamination of oxygen at the interface. Interference fringes due to the strain induced by a 0.01 monolayer between the substrate and epilayer could be observed. We were able to calculate Cu$K\alpha_1$ rocking curves in good agreement with the measured data. Further it was possible to correlate the SIMS and XTEM-measurements with the HRXRD measurements. On the other side, we also compared the RBS channelling measurements with the HRXRD data and simulation. The RBS channelling measurements were very sensitive on the surface, but not on the interface.

We performed also triple axis measurements as described in the paper of P. Fewster [3] for some of the samples. High primary beam intensity is necessary to obtain the information needed for a characterisation of the crystalline quality in the tail of the Bragg peak.

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X-RAY EXAMINATION OF SiC MONOCRYSTALS

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SiC monocrystals grown by the Acheson or modified Lely method have been examined with different X-ray diffraction techniques. These samples belonged typically to the hexagonal polytyp 6H (Ramsdell notation).

The following properties were investigated:

- lattice constants c and a with the BOND method,
- lattice defects (bending, deformation, mosaicity) by measuring the rocking curve with one- or two-crystal diffractometers,
- defect distribution or location by LANG topography,
- dependence of double reflections (Umweganregung) on degree of the perfection.

The most important result is that the lattice constants change only very little between different samples and measuring points on a given sample (with deviations lower than 10 ppm for the lattice constant c). At the same time however the other diagnostic techniques showed a greater lattice distortion. In the X-ray diagnostics of SiC (e.g. lattice relaxations, stacking faults), the weak reflections, forbidden by the space group (e.g. 00.3) or nearly forbidden by the structure (e.g. 00.4), are particulary important. The double reflection effect is considered in this connection.

This work has been supported by the Deutsche Forschungsgemeinschaft (Sonderforschungsbereich 196, project C01).

WHITE BEAM SYNCHROTRON TOPOGRAPHIC ANALYSIS OF MULTI-POLYTYPE SiC DEVICE CONFIGURATIONS

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White beam synchrotron topographic analysis of SiC device configurations comprising various polytypes has been carried out. The devices, pn junctions of area 1 or 3mm^2, were fabricated via CVD epilayer growth of nominally 3C- or 6H-SiC[1], on 6H-SiC substrates grown by either the Physical Vapor Transport technique[2] or by the Lely technique[3]. Detailed analysis of diffracted intensities from the different device areas will be presented. This analysis indicates that those devices which were nominally 3C structure also had some 15R structure present. Depth profiling, utilizing variable penetration depth, grazing-incidence geometries, was used to reveal a layer-type configuration in these multi-polytype epilayers. The presence of "double positioning boundaries" (dpb's)[4] in the 15R layers was also revealed. Superscrew dislocations, present in the substrates grown by physical vapor transport, were invariably observed to be clearly replicated in the 6H epilayers but in most cases not in the 3C/15R epilayers. Growth spirals were occasionally observed in the 6H epilayers at the outcrops of the screw dislocations. The potential implications of the observed defect structures on device performance will be discussed.

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WHITE BEAM SYNCHROTRON TOPOGRAPHIC STUDIES OF DEFECT/GRAIN BOUNDARY INTERACTIONS IN LARGE-GRAIN, POLYCRYSTALLINE ICE

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Synchrotron white beam X-ray topography has been used to conduct *in situ*, low temperature studies of defect/grain boundary interactions in large-grain, polycrystalline ice. The generation of faulted and unfaulted interstitial dislocation loops as a function of temperature was monitored and the influence of grain boundaries on the distribution of these loops was investigated. Results are discussed in the context of diffusion mobilities on the basal plane and the relative orientation between the basal plane and the grain boundary plane. Dislocation generation mechanisms under applied compressive stresses were also investigated *in situ* using a specially-designed compression stage. Grain boundaries were found to act both as effective sources and as strong obstacles to dislocation motion. Generally, the basal slip system with the highest Schmid factor was found to be the most active in polycrystalline ice whereas grain boundary orientation relative to the loading direction seemed unimportant. The generality of these observations is discussed.

WHITE BEAM SYNCHROTRON TOPOGRAPHIC STUDIES OF DEFECTS IN 6H-SiC SINGLE CRYSTALS

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White beam synchrotron topography studies have been carried out on 6H-SiC single crystals grown by both physical vapor transport and by the Lely method. In the crystals grown by physical vapor transport, two general types of dislocation were observed, long straight screw dislocations with Burgers vector equal to a multiple of the c lattice parameter, and basal plane dislocations with Burgers vector $1/3 < 11\bar{2}0 >$. Detailed mapping of misorientations around these screw dislocations achieved by recording topographs at a series of specimen-film distances is presented. The contribution of wavelength dispersion to these dislocations images is discussed. Optical microscopy and SEM analysis reveal that those screw dislocations with the largest Burgers vectors have hollow cores as predicted theoretically by Frank.[1] In addition, when these dislocations emerge on surfaces oriented close to (0001) growth spirals[2] can be clearly observed. Those spirals associated with the dislocations exhibiting hollow cores are often observed to have several "spokes", indicating the presence of a closely spaced group of dislocations. Single "spoke" spirals are also observed associated with dislocations exhibiting no discernible hollow core

Observations carried out in various diffraction geometries indicate that the basal plane dislocations appear to emanate from the superscrew dislocations. A possible mechanism whereby a screw dislocation can generate a dislocations loop with an orthogonal Burgers vector during the high temperature growth process will be presented

Comparison will also be drawn between defect structures observed in epilayers with those in the underlying substrates. Of note is the absence of basal plane dislocation generation in the lower temperature epilayer growth process. This observation is discussed in the light of the mechanism for basal plane dislocation generation in the high temperature bulk growth process

In the Lely crystals studied, basal plane dislocations with Burgers vector $1/3 < 11\bar{2}0 >$ were again observed, as well as basal plane stacking faults stacking faults, with associated partial dislocations

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A NEUTRON DOUBLE-CRYSTAL DIFFRACTOMETER WITH A VARIABLE ANGULAR RESOLUTION

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Recently a version of a neutron double-crystal diffractometer with a variable angular resolution was proposed [1]. With this instrument the scattering diagram of a sample positioned between both the crystals can be registered as a whole by a position sensitive detector.
Such an instrument was installed at the research reactor BER II at the Hahn Meitner Institute Berlin (Berlin Neutron Scattering Center). Fig. 1 shows its scheme.

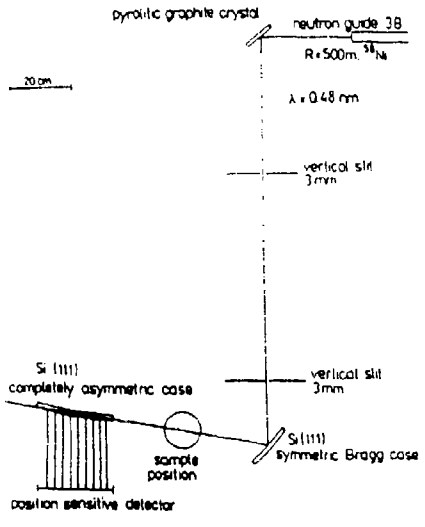

Fig.1 Scheme of the device

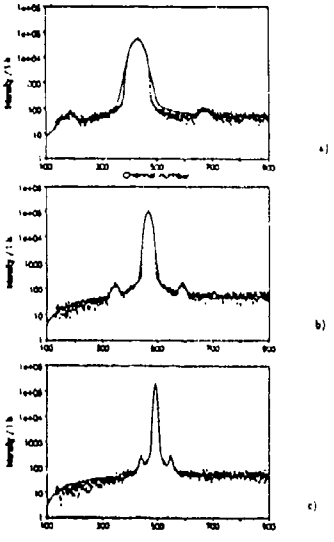

Fig.2 Diffraction pattern from a holographic grating for second crystal bending radii a) 60.5 m, b) 30.3 m, and c) 13.4 m.

The main components of the device are two perfect crystals which can be elastically bent up to a radius of 10 m with an axis parallel to the scattering plane. The second (analyzer) crystal reflects the beam only in the case that its net planes are parallel to the net planes of the first crystal within an accuracy of 0.1 seconds of arc. Therefore, the bent crystal fulfills the Bragg condition only at a small part of its length. If a sample deflects neutrons (e.g. by small-angle scattering, diffraction or reflection) then these parts of the beam are reflected at different crystal places depending on the scattering angle. The intensity distribution is registered by a one-dimensional position sensitive detector.
It is possible to investigate scattered neutron intensity in the angle range from some seconds of arc to 50 minutes of arc. For small-angle scattering it means a Q range from $1 \cdot 10^{-4}$ to $2 \cdot 10^{-1}$ nm^{-1}.
The Fig. 2 demonstrates the function of the device for diffraction from a holographic (refractive index) grating if the angular acceptance is controlled by choosing the bending radius.
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MEASUREMENTS OF THE STATE OF POLARIZATION IN THE FORWARD DIFFRACTED BEAM

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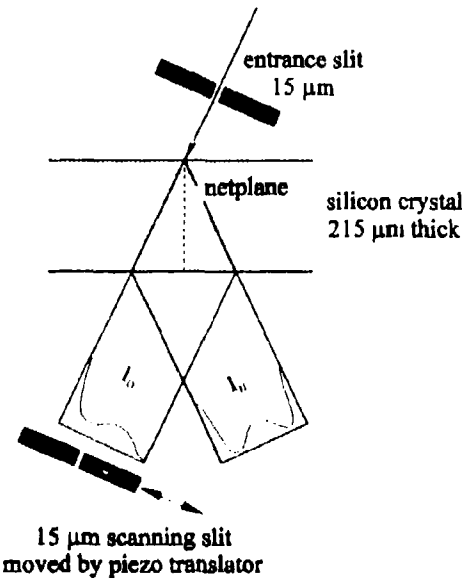
The aim of the experiment is to realize an experimental arrangement for producing elliptically polarized X-rays in the straight forward path way in LAUE case. /1/
Calculations based on the TAKAGI-equations of the dynamical theory of X-ray diffraction /2/ predict the occurence of elliptically or circularly polarized radiation, respectively, inside narrow regions of the transmitted and the reflected beam in the LAUE-case.
By coherent excitation of the σ– and the π– component of the dielectric displacement vector within a perfect crystal, it is possible to produce a circularly polarized beam. The vector of the dielectrical displacement of the linearly polarized incident beam and the diffraction plane inclines an angle of 45°.
The figure shows the BORRMANN-fan of the symmetrical 220-reflection in case of a spherical incident wave. The incident wave comes into the crystal through a fixed narrow slit. The intensity distribution on the back site of the crystal is measured by scanning a second slit.
To analyze the state of polarization of the forward diffracted beam in the narrow scanning-slit region on the back site of the crystal, the integral intensity of a single crystal BRAGG-reflection of $2\Theta_B = 90°$ has to be measured at different azimuthal positions of the analyzer realized by rotating the analysing crystal around the incident beam.



Scheme of the experimental arrangement

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ELECTRONIC MEANS OF COMMUNICATION

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New tools are now available which allow fast communication and dissimination of information between scientists.

The International Union of Crystallography has established a database of crystallographers which is accessible through the networks. Very soon journals will be sent electronically. Most synchrotron facilities have now installed the newest hypermedia hypertext facility WWW (World Wide Web) wnich allows from any terminal to recover an information as text, graphics, image and to print it locally. As a Community the scientists working in High Precision Diffraction and Topography will have to use these new tools. A distribution list is already ready for use. New services may be installed at wish.

This poster will present some examples of these new facilities. If feasable, so me simple demonstrations will be shown.

HRXRD INVESTIGATION OF THE MAIN FEATURES OF THE PROCESSES OF THE EPITAXIAL GROWTH AND DEFECTS GENERATION IN HETEROSTRUCTURES

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There is currently an extremely strong interest in advanced electronic structures in the investigation of processes of epitaxial growth and defect generation because of their great influence on the physical properties of heterostructures. Clearly, the physical properties of heterostructures are extremely affected by different structural defects. The existence of the strong correlation between the growth conditions and crystal perfection of heterostructures is the main reason for further investigation of these processes by means of HRXRD techniques, which are effective non-destructive methods for characterization of these materials.

The interference X-ray diffraction method [1,2] which has an extremely high sensitivity to the crystal perfection and space resolution (up to one monolayer for definite type of heterostructures [3]) and differentional one [4] allow to determine the structural parameters of any type of heterostructures including multilayer and QW ones, allow to study the process of epitaxial growth including its initial stage and the processes of elastic strain relaxation and defects generation

The results of our investigations of lattice matched InGaAsP QW grown by LPE and lattice strained InGaAs QW DH grown by MOCVD technique show the existence of strong correlation between epitaxial growth conditions and the process of defect generation. Those results allow to suggest the qualitative model of the processes of epitaxial growth, elastic strain relaxation and defect generation in heterostructures. It was shown that the main type of structural defects in heterostructures are the own growth defects. The main reason for the beginning of the process of defect creation is an appearence of elastic strain under the epitaxial growth. This is a result of 1)imperfect growth conditions (for lattice matched heterostructures), which leads to the appearence of the stretched elastically strained intermediate layer near the first interface or 2) lattice mismatch which initially exist in lattice mismatched heterostructures

Preliminary investigations of mismatched heterostructures grown by MOCVD technique show that the initial stage of the epitaxial growth affects strongly on the process of elastic strain relaxation and results in two different types of crystal structure of the strained layers. It was also shown that under the perfect growth conditions the critical thickness of such layers may be exceeded three to five times

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A NEW INTENSIFIED CCD–CAMERA FOR X–RAY APPLICATIONS

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Compared with the widely used method of recording x–ray images as radiographs on photographic emulsion, electronic cameras have the advantage of saving the wet chemical process of film development. Moreover the digitized data are immediately available for image or data processing purposes. However, the resolution of an electronic camera system is worse than that of film material.

The camera presented here uses the "indirect" method: x–ray quanta (8 keV) are converted to visible light in a phosphor screen, this is intensified in a one–stage microchannel plate image intensifier and detected with a cooled slow–scan CCD camera with a 1k x 1k chip and $(19\ \mu m)^2$ pixel size. The camera is designed to gain high spatial resolution. The optical connections consist of fibre optics. Taking into account the optical transfer one CCD pixel corresponds to $(8.8\ \mu m)^2$ on the phosphor screen. Furthermore, the detection of single x–ray quanta is intended. For these purposes the charge which is generated in the CCD pixels during exposure is *not* accumulated on chip. Instead a numerical algorithm for the digitized data will be used taking advantage of the fast signal processor on the frame grabber board.

Technical information about the camera and results of tests concerning resolution and quantum efficiency will be presented.

IN SITU X-RAY INVESTIGATIONS OF THE HIGH TEMPERATURE BEHAVIOUR OF STRAINED $Si_{1-x}Ge_x/Si$ AND $Si_{1-y}C_y/Si$ HETEROSTRUCTURES

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We investigated RTCVD- and MBE-grown pseudomorphic $Si_{1-x}Ge_x$ ($0.15<x<0.35$) and $Si_{1-y}C_y$ ($0.008<y<0.016$) thin layer on Si(100) substrate at anneal temperatures up to 1000 °C. To measure *in-situ* the variation of the epilayer lattice constant, a conventional X-ray powder diffractometer with high temperature attachment in combination with a high power X-ray generator was used. The measurements were carried out by successive $\theta/2\theta$-scans of the Cu K_α radiation (400)-reflexion during post growth annealing, with a time resolution of about 5 minutes. From the decrease of the angular distance between substrate and layer reflexion, the strain state of the system can be obtained. The results given by this low resolution powder diffractometer method were compared *ex-situ* with high resolution double and triple crystal diffractometer measurements.

In the compressive strained $Si_{1-x}Ge_x/Si$ system we could observe strain relaxation which is explicable by the introduction of misfit dislocations at the interface. At temperatures about 1000 °C an additional effect, the interdiffusion of Si and Ge and thus the decrease of the Ge concentration inside the layer, enhances the relief of the strain. Both effects were separately measurable by successive measurements of symmetrical (400)- and asymmetrical (311)-reflexion during isothermal annealing, and the diffusion coefficient was determined in its dependence on the Ge content.

On the other side, in the tensile strained $Si_{1-y}C_y/Si$ heterostructures at temperatures beyond 800°C, the nucleation and diffusion controlled growth of SiC nanocrystals was found to be the dominant mechanism decreasing the amount of C atoms on substitutional sites and thus relieving the strain in the layer. The decrease of the carbon content during isothermal annealing exhibited exponential behaviour, with a time constant showing an Arrhenius-like temperature dependence. From this result, we determined an activation energy corresponding to the known activation energy for the diffusion of substitutional carbon in silicon.

INVESTIGATION OF THE CRYSTAL QUALITY OF InAs/GaAs HETEROSTRUCTURES BY HIGH RESOLUTION X-RAY DIFFRACTION

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InAs layers have been grown by conventional Molecular Beam Epitaxy on (001) oriented GaAs substrates; a 0.2 μm thick GaAs buffer layer was always interposed between the substrate and the heterolayer. The growth temperature T_g was varied from 350 to 500°C and the layer thickness t_l was 2 and 6 μm.

The crystalline quality of the specimens have been investigated by a high resolution Philips diffractometer equipped with a two-crystal four-reflection monochromator (Ge, 220); the Cu $K\alpha_1$ radiation and the symmetric 004 reflection were used. The assumption was made that the full width at half maximum β of the layer Bragg peak is determined solely by threading dislocations; then the concentration of the threading dislocations ρ was calculated by the expression $\rho=(\beta/3b)^2$, where b is the length of the Burgers vector.

It has been found that β strongly decreases by increasing T_g. The values β=492, 347, 319, 275 arcsec were indeed measured for 2 μm thick layers grown at T_g =350, 400, 450, and 500°C. Assuming that the threading dislocations are of 60° type, their concentration ρ changes from about 3.5×10^8 to 1.1×10^8 cm^{-2} by increasing T_g from 350 to 500°C.

A strong improvement of the crystal quality has been observed at each growth temperature by increasing t_l. For example, at T_g=350°C the values β=492 and 268 arcsec (corresponding to $\rho=3.5\times10^8$ and 1.0×10^8 cm^{-2}) were calculated for t_l=2 and 6 μm respectively, whereas at T_g=500°C the values β=275 and 154 arcsec (corresponding to $\rho=1.1\times10^8$ and 0.3×10^8 cm^{-2}) were measured.

It can be concluded that the crystal quality of the InAs heterolayers improves by increasing the growth temperature, possibly due to the enhanced surface mobility of In, and by increasing the layer thickness as a consequence of dislocation recombination and annichilation mechanisms.

HIGH RESOLUTION X-RAY DIFFRACTION STUDY OF MOVPE InGaAs/InP SUPERLATTICES

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30 period InGaAs/InP nearly lattice matched superlattices have been grown by Metal Organic Vapour Phase Epitaxy on (001). Two different specimen series were prepared: in the first the InGaAs well thickness was kept fixed at 3 nm, while the InP barrier thickness was varied from 15 to 3 nm; in the second the barrier thickness was 2.3 nm and the well thickness varied from 9.2 to 2 nm.

The structural properties of the superlattices have been investigated by high resolution X-ray diffractometry using a Philips diffractometer equipped with a two-crystal four-reflection monochromator (Ge, 220); the Cu $K\alpha_1$ radiation and both the symmetric 004 and 002 reflections were used. The first reflection permits a better separation between 0-order and substrate peaks, whereas the second evidences the modulation of the satellite peak intensity which is related to the thickness of the thinnest layer in the period. The experimental curves were fitted using a computer program based on the dynamical diffraction theory.

When the barrier is much thicker than the well the superlattice has a high structural quality. Only the presence of thin intermediate layers has been assumed in order to fit the experimental data: at the lower interface of the well (i.e. from InP to InGaAs) about 3 monolayers of InGaAsP have been introduced, whereas at the upper interface (i.e. from InGaAs to InP) about 4 monolayers of InAsP has been considered. The structural quality strongly worsens with barrier thickness decreasing; when the barrier is as thin as 4-5 nm, only a few satellites are detectable, their shape is asymmetric and the intensity low.

When the well is much thicker than the barrier the structural quality is as good as in the reverse case. The experimental curve could not be fitted in a satisfactory way, although the period, the average well composition and the well-to-barrier thickness ratio were found to be in good agreement with the project parameters. The quality of the superlattices has been found to worsen by decreasing the well thickness. When the barrier was 2.3 nm and the well 2.0 nm, no satellites were detectable.

HIGH-RESOLUTION X-RAY SPECTROSCOPY USING A JOHANN SPECTROMETER IN VERTICAL GEOMETRY

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An alternative scheme of the Johann spectrometer, which takes advantage of the divergence of the radiation in the plane perpendicular to the plane of incidence [1] is used for x-ray spectroscopy. Compared with other well known techniques (for example single crystal or the conventional Johann geometry), it provides at the same time high spectral resolution, luminosity and 1-D spatial resolution. The unique features of this instrument are used for two purposes:

1. Excitation-energy dependence of X-ray emission line shapes

Recently, high-precision measurements of spectral line shapes of x-ray emission lines using single- and double-crystal-spectroscopy are published [2,3]. The measured line-shape was interpreted in terms of an empirical Lorentzian model [2], respectively on the basis of Dirac-Fock-calculations of "spectator hole" transitions [3]. Experiments with a vertical Johann spectometer at the monochromator beamline of HASYLAB Hamburg were carried out to show the influence of different excitation energies near the K-absorption edge of iron on the spectral line shape of the x-ray fluorescence lines.

2. High-resolution spectroscopy of laser produced plasmas

In plasma diagnostics the above mentioned properties of the vertical Johann geometry are necessary for detailed studies of plasma parameters. In experiments at the Max-Planck Institute of Quantum optics in Garching laser pulses were focused to massive KCl targets to create a high density Cl plasma. The Heα-group of He- and Li-like ions were recorded at two different directions of observation (nearly parallel and with an angle of 45° to the target surface). The time integrated spatial distribution of the main plasma parameters, Doppler shift of the emission lines, gradients of the ion velocity and opacity effects on the line position were determined.

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USING OF TOPOGRAMS IN LENSLESS X-RAY OPTICS

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In the report the possibility of usage of perfect crystal X-ray topograms as focusing optical elements is considered. It is shown that:

(i). The plane wave topogram of a crystal with exit surface of spherical shape is a system of interference fringes coincident with Fresnel zones. The zone sizes and their quantity can be variable over the wide range of values depending on X-ray wavelength and crystal type. Such a topogram may be used as zone plate for soft X-ray radiation;

(ii). Using the section topogram of a plane - parallel crystal one can obtain a magnified visible image of the X-ray source. To do this the thin crystal section topogram should be illuminated by light wave which being diffracted by the system of pendellozung fringes is focused forming the X ray source image. The focal length, magnification and image diffraction sizes are calculated.

XRD CHARACTERIZATION OF IMPLANTATION DAMAGE IN InSb REDUCED BY MAGNETIC FIELD

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Ion implantation plays important role in device fabrication in modern semiconductor technology. Since the radiation induced damage influences the electronic properties, post implantation treatment is required to remove it. It has been shown recently that a weak permanent magnetic field (PMF) could reduce significantly the implantation damage in $Hg_{0.8}Cd_{0.2}Te$ crystals[1]. Analogous effect has been observed in InSb crystals implanted with In atoms at 320kev with a dose $1 \cdot 10^{13} cm^{-2}$. In this work the structural perfection of the implanted layers was characterized by triple axis and grazing incidence X-ray diffraction carried out on Philips MRD diffractometer[2]. The $\theta : 2\theta$ scans for samples implanted in magnetic field and in the absence of the magnetic field in comparison with the pre-implanted sample showed that the surface layer undergoes a lattice expansion. A smaller amount of strain resulted in the case of the sample treated in PMF. The comparison of the rocking curves for these samples also showed the shift of the low-angle side of the Bragg peak characteristic of lattice expansion in the implanted layer, which was more pronounced in the case of magnetic field absence. However, for the sample implanted in PMF, more diffuse intensity is observed in the vicinity of the Bragg peak implying increased tendency for the formation of extended defects and/or their depth distribution closer to the surface of the crystal. The swelling of the implanted surface layer was also observed in the intensity patterns of asymmetric reflections measured from a number of planes inclined to the surface. In addition, rocking curves from the $\{\bar{2}\bar{2}4\}$ planes perpendicular to the surface were measured under incident and exit grazing angle conditions. The patterns showed features similar to the ones observed in normal incidence. The scattering is shifted to the low-angle side of the Bragg peak indicating a positive strain of the lattice planes, which is smaller for the sample implanted in PMF, while a higher intensity around the Bragg peak for this sample points to a larger size of the defects and/or their localization closer to the surface. The interpretation of the Rutherford backscattering channeling spectra measured on these samples supports the results of X-ray diffraction.

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Residual and thermal strain investigation of ZnS epitaxial layers grown on [100]-GaAs by vapour phase epitaxy

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In this work, ZnS epitaxial layers grown by chemical vapour epitaxy on [100]-oriented GaAs substrates are investigated by x-ray diffraction and secondary ion mass spectroscopy (SIMS).

The residual strain status of the *as-grown* samples was determined by high resolution double crystal x-ray diffraction measurements. Eleven diffraction curves were recorded in the vicinity of the (400), (422) and (531) Bragg reflections in different diffraction geometries and for several azimuth angles. The analysis of the experimental data was performed by using a general model which relates the angular distances between diffraction peaks and strain tensor components in 2nd-order approximation. This model considers the lowest crystallographic symmetry (triclinic) for the lattice distortion of a cubic unit cell. Our results indicates that the crystallographic symmetry of the distorted ZnS unit cell is orthorhombic which is explained by an anistropic dislocation density distribution in the interface plane.

In order to determine the strain contribution due to the differences in the thermal expansion coefficients between ZnS and GaAs (thermal strain) the temperature variation of the residual strain was measured between 25 and 650 °C by using a single crystal x-ray diffractometer. In this work, we determine experimentally for the first time the thermal misfit between ZnS and GaAs and the ZnS thermal dilatation coefficient.

Finally, the chemical composition of the ZnS/GaAs interface for the *as-grown* and the annealed samples was measured combining triple crystal x-ray diffraction and SIMS techniques. An interdiffused layer was revealed at the heterointerface which presence was found to be not related to the strain relief process, but it is probably caused by the intermixing of the atoms at the early stage of the epitaxial growth.

DIAMOND PHASE PLATES FOR X-RAY POLARIMETRY AT THE ESRF

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Low emittance third-generation storage rings, like the ESRF, provide an optimum condition for the use of perfect crystal phase plates combined with planar undulator sources. Their low angular and spatial distributions in both the horizontal and vertical directions are suitable to an excellent performance of diamond crystals as quarter-wave plates (QWP) and half-wave plates (HWP). Recent experiments performed at ID10 (Troika Beamline) at the ESRF confirm 99% and 97% efficiencies of diamond phase plates in the transformation of horizontal linear polarization into circular polarization and linear polarization in the vertical plane respectively. Furthermore, the successive use of two QWP allowed the production of linear polarization in any desired direction and the complete characterization of the polarization state of a circularly polarized beam within 1%. The diamond plate was set in an asymmetric Laue transmission geometry at 9 keV, allowing a transmission factor of 0.4. Preliminary calculations indicate that such phase plates with suitable thicknesses could be efficiently used in the energy range from 4 to 17 keV.

CHARACTERIZATION OF MONOLAYERS AND MULTILAYERS BY A CONVENTIONAL POWDER DIFFRACTOMETER WITH SPECULAR REFLECTIVITY

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Thin films are generally analyzed by using synchrotron radiation with highly collimated beams or line focus rotating anode X-rays in connection with channel cut monochromators. Improvements of commercially available powder diffractometers permit today to perform reflectometry experiments also with conventional x-ray radiation. The great advantage is the easy access in basically every laboratory. The investigations reported here have been performed on a Siemens D 5000 powder diffractometer with graphite monochromatized Cu-Kα radiation. Information about thickness, density and roughness were obtained by least-squares refinement methods assuming different models.

We will report on the analysis of a 500 Å Pt monolayer on a Si (110) substrate. First results revealed a deviation between observed and calculated reflectivity at higher angles. Assuming a thin oxygen surface layer (14 Å) on Pt led to a complete match of both curves. Oxydation of the top layer is not uncommon. Furthermore we could demonstrate that a misalignment of the film surface of $\pm 0.2°$ resulted in no significant change of thickness ($< 1\%$), of density (8%) and of roughness ($< 1\%$) calculated from the observed data. This insensitivity is quite surprising and is another advantage of such an experimental setup. In a second example the dependence of layer-thickness from sputtering at different argon pressures will be shown for two 300 Å Pt films.

An important application is found in the investigation of multilayers, consisting of pairs of bilayers. Bragg reflections and multiple Kiessig interference fringes can be observed in X-ray reflectometry yielding thickness, density, surface roughness and especially diffusion. As an example we will discuss a Pt\Co multilayer, where we could observe diffusion of Co into the Pt-layer.

DIFFUSE X-RAY SCATTERING FROM MISFIT DISLOCATIONS AT SEMICONDUCTOR HETEROINTERFACES

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ABSTRACT

X-ray diffuse scattering in (001) InGaAs/(AlGa)As high electron mobility transistor structures was observed for structures whose InGaAs channel thickness exceeded the critical thickness. The diffuse scattering was determined to originate with misfit dislocations, as confirmed by plan view transmission electron microscopy (TEM). Diffuse scattering was measured using triple axis x-ray diffraction, which showed that the diffuse scatter was sensitive to the density and direction of the misfit dislocations. Using TEM for calibration, we determined that the diffuse scattered intensity is directly proportional to the dislocation density. For samples with misfit dislocations along only $[\bar{1}10]$, diffuse scattering was confined to a crystallographic direction which was perpendicular to the misfit segments, but for samples with thicker InGaAs layers - and misfit segments in both <110> directions - diffuse scattering existed in a more random distribution around the reciprocal lattice point. We also found that the observation of directional diffuse scattering provided a more sensitive means to detect misfit segments than other commonly used techniques. The presence of an orthogonal array of misfit dislocations impaired device performance, but the best device performance occurred in the samples with a one dimensional array of misfit segments, demonstrating that this technique is useful for predicting device performance.

THE DETECTION OF PRECIPITATES BY X-RAY TOPOGRAPHY

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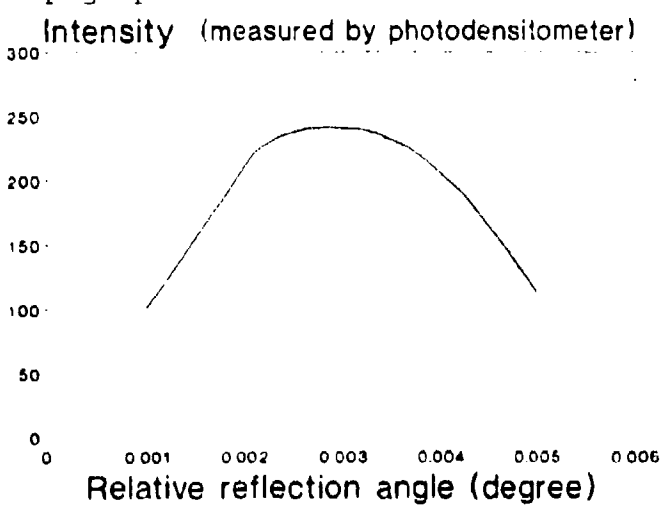
Nowadays, substrates of semiconductor devices are mostly dislocation-free single crystal slices of silicon, gallium arsenide, etc., but nevertheless they may contain precipitates. Also technological processes i.e. doping by adequate atoms followed by heat treatment may result precipitates. To determine whether construction or dilatation happened in the lattice surrounding the precipitates is essential and also at least a rough estimate of the value for this contraction or dilatation is desirable.

For solving this problem the "method of exposure series" (MES) was used. By turning the well oriented sample around its vertical axis by series of adequate angles and at each setting, a topograph was recorded to the same film. From the intensity values diffracted at each position by the substrate its rocking-curve can be derived (see figure).
Comparing the pictures, where the precipitates reflecting with maximum intensity to those pictures, where their enviroment (substrate) are in ideal reflecting position angles, it's possible to decide whether the faults cause local contraction or dilatation in the lattice. Getting to a better estimation, this procedure must be done repeatedly for reflections of medium intensity.
In reality, the situation is more complex, the sample has some curvature and/or contains growth circles, causing an intensity fluctuation between different regions of the topographs.



As an example we have investigated the precipitates created in a (100) silicon slice by doped with B (10^{18}-10^{19}/cm^3) lying in a 1 μm thick surface layer. Due to the curvature of sample in different regions the precipitates appear as dark spots, light spots or partly dark and light spots.

MEASUREMENT OF INTERFACE ROUGHNESS IN A SUPERLATTICE OF DELTA-BARRIERS OF ALUMINIUM IN GaAs USING HIGH-RESOLUTION X-RAY DIFFRACTOMETRY

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Delta-doping, or the confinement of dopants to a single atomic plane, has useful applications for electronic devices. A study of Si δ-doping in GaAs has shown that if the growth temperature is low to prevent diffusion and surface segregation, and the arsenic flux is maintained during deposition so that dopant atoms are located on Ga sites only, then it is possible to confine the dopant atoms to a single atomic layer, with a spread of no more than 1½ monolayers (ML)[1]. However, once the Si coverage reaches ½ ML, there is a loss of electrical conductivity, implying that there are local distortions of the lattice sites and/or changes in the charge state of the Si in the δ-layers[2]. In an attempt to understand this, a comparison has now been made with Al δ-barriers since Al, unlike Si does not have the complications of amphoteric behaviour and bistability in lattice location.

Molecular beam epitaxy was used to grow superlattice structures on (001) GaAs substrates at 400°C with the arsenic flux maintained during deposition of aluminium. Samples consisted of 20 to 100 δ-layers of 1.0, 0.5 and 0.1 ML of aluminium, each separated by 500Å GaAs. A Philips high-resolution, triple-axis diffractometer with a 4-reflection Ge 220 monochromator and a 2-reflection Ge 220 analyser was used to map reciprocal space close to the 002 Bragg reflection. Information obtainable by high-resolution x-ray diffractometry includes the total aluminium concentration, average layer thicknesses, variation in superlattice period, spreading of δ-layers perpendicular to the surface and interface shape parallel to the surface. The superlattice period is determined from the spacing of satellite peaks visible on either side of the average layer peak. Variations in the period cause broadening of the satellites while interface grading changes their integrated intensities.

For Al in GaAs, the structure factor sensitive 002 reflection gave the most highly visible satellites. In one sample, more than 30 satellite peaks were visible on either side of the main Bragg peak. Comparison with dynamical simulations resulted in best-fit models for the structures of 1.4, 0.7 and 0.25 ML of AlAs and 472Å GaAs, with a 2% period variation. Simulations also showed that there was no spreading (interface grading or roughness) beyond 2ML (6Å). In conclusion, high-resolution x-ray diffractometry can be used to characterise superlattice structures with δ-layers less than 2ML in thickness. The Al δ-barriers behaved as expected, showing that in Si δ-doping, some change must occur in the charge state and/or local lattice distortion which does not occur for Al.

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THE RELAXATION LINE IN RECIPROCAL SPACE – THE USE OF A MODEL

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Reciprocal space mapping is a powerful method for the detailed structural characterization of even nonperfect crystalline layers. This will be demonstrated on the example of epitaxially grown II-VI heterostructures. Thereby especial attention will be attached to the model of the relaxation line. It describes the effect of the elastic strain and its relaxation in an epitaxial layer on the distributions of the scattered intensity in reciprocal space. This effect can be a shift of the maximum or/and a broadening of the scattered intensity along this line the direction of which depends on the elastic constants of the layer material and on the order of the Bragg reflection.

Rocking curves and the reciprocal space maps of Bragg reflections performed in symmetrical scattering geometry are affected in a similar way by gradients of the relaxation state as well as by variations of the chemical composition. However, for reciprocal space maps of asymmetrical Bragg reflections the directions of the broadenings caused by these two gradients are essentially different. Knowing the direction of the relaxation line, this enables one to distinguish the two kinds of gradients. This is demonstrated for two examples: a HgCdTe layer showing a variable Cd content, and a CdMnTe layer with a gradient of the strain state the latter has been proved indirectly by photoluminescence.

The relaxation line is useful for the description of thermoelastic strain in heterostructures, too. This strain is caused by different linear thermal expansions of the layer and substrate materials during cooling down a heterostructure from growth to characterization temperature. Thermal induced strain states have been systematically observed for HgSe epilayers on ZnTe buffers on GaAs(001) substrates. Despite of the low HgSe growth temperatures of only 110°C, the thermoelastic strain is clearly visible in this epitaxial system due to the large differences in thermal expansion coefficients of the materials concerned. The effect of thermoelastic strain can be influenced by varying the temperature either of the growth or of the characterization which will be demonstrated.

NEW X-RAY REFRACTOGRAPHY FOR NONDESTRUCTIVE INVESTIGATION OF ADVANCED MATERIALS

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X-ray refractography is an unconventional x-ray scattering technique which has been developed and applied to meet the actual demand for improved nondestructive characterization of high performance composites, ceramics and other low density materials and components. It determines the amount of inner surfaces and interfaces of nanometer dimensions due to the short X-ray wavelength of ~ 0.1 nm. We can measure the pore size of ceramics without destroying the structure by cutting or polishing for microscopy techniques.

Scanning refractometry visualizes the projection of fibre nanometer debonding at 20 μm spatial resolution. It performs an additional sensitivity to fibre orientation and has turned out to be an effective tool for determining interfaces in composites. Mean values of the fraction of debonded fibres are measured to 1% precision within a second. This is much more precise and faster than any mechanical testing could be, a desirable situation when processing is to be optimised or delicate components have to be evaluated. The method requires a (commercial) small angle x-ray camera, a standard fine structure x-ray generator, X-ray detector and computerized manipulator. X-ray refraction can be understood by analogy to the well known refraction of visible light which is governed by Snell's law. It has not been exploited so far in material science.

Scanning a sample in the X-ray beam and detecting the refracted intensities for each position under a fixed scattering angle of typically one minute of arc provides the data for a two-dimensional computer image of the inner surface distribution. The high scattering intensities provide the possibility of high scanning speed and imaging within several minutes. Examples of analytical investigations and nondestructive inspections by scanning will be presented. Applications to various heterogeneous materials including CMC, HT superconductors, monolithic ceramics, injection moulding composites, ferrofluids, video tapes, filters, paper, x-ray films and pigments were successful.

Precision determination of lattice constants and Poisson ratios in the system AlAs - GaAs

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Using Vegard's rule to calculate compositions from X-ray results, knowledge of the elastic constants and the lattice parameter of AlAs are prerequisite to accurate evaluations. In order to resolve discrepancies in the literature we performed X-ray diffraction and Brillouin scattering experiments on a series of $Al_xGa_{1-x}As$ films. These films with nominal Aluminum compositions of 20%, 40%, 70% and 100% and thicknesses between 1 µm and 10 µm have been grown on 3-inch GaAs(001) wafers by MOCVD and MBE.

Brillouin scattering was performed with a plane tandem triple-pass Fabry-Perot interferometer modified to operate in the near infrared where the low optical absorption allows to measure bulk properties. Using three different light-scattering geometries it was possible to obtain all three elastic constants for each sample and therefore determine the Poisson ratios to a high precision.

X-ray diffraction measurements were performed using a high resolution diffractometer equipped with accurate angle reading (<1") and a full circle Eulerian craddle. The measurement procedure is based on the Bond technique, it comprises measurements within several angular ranges belonging to reflections of one crystallographic zone. Since the zone axis is set closely parallel to the Ω-axis prior to the measurements, no readjustment of the sample orientation is required, when moving from one reflection range to the next within a given zone. For each sample several zones have been measured. Care has been taken to minimize known sources of systematic errors (wavelength calibration, temperature stability, refraction corrections). Using symmetric (004, 006) and asymmetric (115, 224, 206) reflections the tetragonal metrices of elastically strained GaAs substrates and AlGaAs films have been determined with good accuracy.

We will present a new fundamental equation relating X-ray data (peak splitting) to composition, compare our results with literature data, discuss sources of error and detail accuracies when performing routine X-ray diffractometry of $Al_xGa_{1-x}As$ films on GaAs(001) substrates.

SIMULATION OF X-RAY SECTION TOPOGRAPH IMAGES OF OXYGEN PRECIPITATES IN SILICON

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As a result of heat treatment during the various processing stages before and during device fabrication, oxygen may precipitate out in otherwise highly perfect silicon crystals. Characterisation and control of the distribution of these defects is of major interest within the semiconductor industry. X-ray section topography is a highly appropriate method for the non-destructive characterisation of such defects and by use of simulation techniques based on the solution of Takagi's equations, the local strain can be determined by matching simulated and experimental images.

We have extended our previous studies of the contrast of spherical defects by incorporation of the effect of surface relaxation. The critical distance from the surface at which these effects become insignificant varies as *ln* C, where C is the deformation parameter in a strain field of the form $\mathbf{u}(\mathbf{r}) = \{C/r^2\}\mathbf{r}$.

During the process of intrinsic gettering, a high density of precipitates is produced in the centre of a silicon wafer with defect-free regions extending typically 30 μm from the surfaces, into which devices are fabricated. Section topography has proved to be a very successful technique for non-destructive monitoring of the depth of these denuded zones. In experimental section topographs, defect images often appear in parts of the topograph apparently associated with the denuded zone. However, variable wavelength double axis topography failed to detect such denuded zone defects [1]. We have simulated images produced by a random distribution of precipitates in the central region only and find good correspondence with experiment. This includes the presence of images in the margins of the topograph. Simulations have been undertaken to determine the defect density at which individual precipitate images can be resolved. As with the surface relaxation condition, the minimum distance at which two precipitates can be individually resolved varies as *ln* C.

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HIGH RESOLUTION X-RAY TOPOGRAPHY STUDY OF PERIODICALLY DOMAIN-INVERTED NONLINEAR OPTICAL CRYSTALS

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Quasi-phase matched second-harmonic generation based on periodically domain-inverted structures has received great attention for novel applications due to the high nonlinear conversion efficiency and the possibility of phase matching arbitrary wavelengths. Techniques to fabricate periodic domain inversion in $LiNbO_3$, $LiTaO_3$, and $KTiOPO_4$, such as proton or ion exchange, electric field poling, and electron lithography, have been developed rapidly. However, it seems that the periodically-poled domain structures fabricated thus far have not been well understood. This lack of fundamental understanding will probably affect adversely the development of reliable and reproducible periodic-poling techniques. Therefore, the establishment of a structural and microstructural understanding of periodic-poling of domains is seen to be of considerable importance and interest.

Using a PHILIPS X-ray diffractometer, we have applied high resolution X-ray diffraction topography combined with reciprocal space mapping for assessment of periodic domain inversion in $LiNbO_3$ and $KTiOPO_4$ crystals. In particular, X-ray reflections, which are highly sensitive to anomalous scattering and strain, respectively, are employed for mapping out the periodically alternating domains and domain walls. Some interesting results will be presented and discussed.

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MICROFOCUS DOUBLE CRYSTAL X-RAY DIFFRACTOMETRY ON III/V-HETEROSTRUCTURES GROWN BY SELECTIVE AREA EPITAXY

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Monolithic Bragg-Fresnel X-ray optics enable a monochromatization and simultaneous focusing of the highly collimated synchrotron radiation beam to a focus of micrometer dimension. In a double crystal arrangement rocking curves can be scanned with this high spatial resolution which allows a detailed study of patterned heteroepitaxial structures.

Our microfocus X-ray diffraction measurements were carried out at the optics beamline of the ESRF using a microfocus double crystal diffractometer in (+n, -m) setting consisting of a Si(111) Bragg-Fresnel lens and the InP(001) based sample. The Si(111) Bragg reflection was set to select 11.6 keV photons from the white X-ray radiation provided by a bending magnet. Perpendicular to the dispersion plane of the Bragg reflection, the cylinder-lens-type Fresnel optics focuses a 100 µm wide beam of 11.6 keV photons to a line focus of 2 µm width. The alignment of the sample pattern relative to the X-ray line focus was performed in this geometry by X-ray topography showing simultaneously the pattern of the sample and the position of the line focus.

With this setup III/V-heterostructures grown by different selective area epitaxy techniques (planar and embedded selective area MOVPE and MOMBE) were investigated. The samples were test structures of InGaAs and InGaAsP layers grown on an InP(001) substrate that was partially masked with SiO_2 fields and laser/waveguide devices laterally integrated on an InP(001) wafer. In order to determine the lattice mismatch close to the boundaries of the layer/oxide and the laser/waveguide boundary, rocking curve scans with micrometer step width were performed. The lattice distortions of the III/V-heterostructures show changes at the boundaries in the range of 5 µm to 100 µm depending on the selected process.

OBSERVATION OF THE PHASE CONTRAST FROM NON-CRYSTALLINE OBJECTS IN THE PLANE WAVE X-RAY TOPOGRAPHY SCHEME.

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The plane wave scheme (n,-n) is used for observation and investigation of micro defects in almost perfect monocrystals. The contrast from these defects may be described in terms of the interference between a spherical wave produced by a crystal defect with a primary plane wave [1]. In our plane wave experiments we found that any perturbations had contributed into an incident wave also become visible after diffraction of said wave in a perfect crystal. This phenomenon was discovered in 1987 when not only ordinary contrast of crystal defects was registered but also some additional contrast due to textile or paper envelope in which the crystal under test had been deposited [2], see Fig. 1.

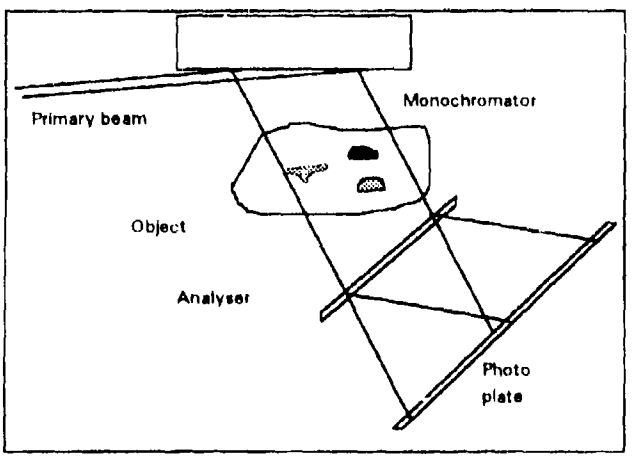

Fig.1. X-ray scheme of PDI method.

The absorption of MoK_α radiation in these envelopes was so small that it could not explain high contrast images on a photo-plate. It also was stated that the rocking curve (RC) became wider after the object deposition into an incident beam. The last crystal in this new scheme became the crystal-analyzer (CA).

The systematic investigations of polymer and biological objects, brought into the double- and triple crystal schemes, discovered the following main features:

- the inner structure of the object is registered;
- the image contrast exists only if CA is within its RC. Only the absorption contrast exists out of the RC;
- the image contrast in transmitted and diffracted beams is almost supplementary;
- the image contrast depends on the CA tie point position;
- the image contrast depended on the plane wave coherence length;
- the image contrast was registered only for objects sufficiently transparent for radiation had used.

The wedge shaped monocrystal was used as CA to investigate the nature of phenomenon. The changes in pendelloesung contrast confirmed that plane wave phase perturbations are the reason of new image. So the new contrast was named the phase-dispersion contrast and the method of structure investigation - the phase dispersion introscopy (PDI).

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THE DIFFRACTED BEAM PENDELLÖSUNG INDUCED BY ULTRASOUND AT THE DEFORMED SILICON SINGLE CRYSTAL

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The classical Pendellösung in perfect single crystals was studied by many authors. The picture of Pendellösung is very sensitive in respect of the value B of inhomogeneous deformation in the crystal [1]. A high-frequency acoustic wave (AW) induces transitions between the sheets of a dispersion surface (DS). The AW violates adiabatic motion of the representative point on the sheets of the DS and strongly decreases intensity I of the diffracted beam [2] for *small* AW amplitude W at the *deformed* crystal. A new Pendellösung induced by the AW at the *deformed* crystal was predicted recently [3]. This "Deformational Pendellösung" (DP) is excited by interference between different paths of the representative point at the DS.The first path is an adiabatic motion from point 1 to 4 at the same sheet of the DS; the second path is absorption of the AW phonon at point 1, an inelastic transition to point 2 at *another* sheet of the DS, adiabatic motion along this other DS sheet up to point 3, emission of the AW phonons and transition to point 4. Therefore the decreasing of intensity Al of the diffracted beam has the form (H-vector of scattering)

$$Al/I \approx -2R(1-R)[1-\cos\Phi],\quad \Phi \sim 1/B,\quad R \approx \exp[-(HW)^2/B].$$

We studied the effects of the transversal AW at the symmetrical Laue diffraction at the deformed silicon single crystal plate for the case of X-ray (MoK_α radiation, (440),(660),(880) reflections) and neutron (λ 0.101 nm, (220)) scattering. We observed the dependence $\Phi \sim 1/B$. The period of these oscillations is in the good agreement with theory (especially for the case of neutron experiments). It is pointed out also that a such "Deformational Pendellösung" must be observed as well for the case of the Bragg geometry at a thick crystal. Application of the DP for the case of X-ray and neutron topography is also discussed.

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THE INFLUENCE OF THE HIGH FREQUENCY ULTRASOUND ON THE PARAMETERS OF DOUBLE-CRYSTAL SPECTROMETERS

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The reasonable combination of high resolution with acceptable intensity I of the diffracted radiations is the one of the problems of double crystals (DC) spectrometers using (especially for the case of neutrons diffraction). Recently [1] it was proposed that the high frequency acoustic waves (AW) excitations with the same frequencies and similar amplitudies W in both crystals- monochromator and analyzer-lead to the serious *simultaneous* improvement of the angular resolution and peak intensity.The mechanism of effect: X-ray or neutron absorbed N>>1 AW phonons in monochromator and resonantly emitted the same N AW phonons in analyzer. Therefore we studied the effects of the AW on the parameters of DC neutron spectrometers. The experiments concerning AW excitation in one crystal-analyser-were realized in Rez. We used DC spectrometer in symmetrical Bragg (monochromator)- Laue (analyzer) geometry. The thick silicon single crystal plates (T=5.0mm) were used. Wave-length of neutrons λ=0.177 nm, reflection $(1,\bar{1},1)$, the angular step - 0.02 sec. The transversal AW frequecy of ν=26.8 MHz propagated in plane of scattering perpendicular to the vector of scattering. Without the AW FWHM of the rocking curve (RC) $2\Delta\Theta_0$=1.94 sec, that is in agreement with theory. The AW excitations transform the Lorentz form of the RC to Gauss, increase FWHM 1.63 times and an area of RC 2.37 times. These results are described by theory. The peak of the intensity I of the RC arrives to saturation and increases 1.65 times, that is don't exceed the theoretical limit 1.7 for the DC Bragg-Laue geometry. The experiments using *simultaneously* AW excitation in *both* crystal (Bragg-Bragg geometry) were realized at the DC DIFRAN at the impulse reactor IBR-2.The silicon single crystal plates (T=6.0mm) were used.The transversal AW ν=26.5 MHz and ν=26.8 MHz (1,3 and 5 harmonics) were excited by $LiNbO_3$ converter and propagated *perpendicular* to the plane of scattering. In all cases the intensities I growth and RC narrowing were observed. For example, at the ν=26.75 MHz intensities gain was 9.3 time and FWHM decreased from 3.4 sec without AW to the 2.8 sec when AW was turned on. The background-to-peak relation increased 2 times. Some details of these very strong AW effects are not comletely understand. Their interpretation is discussed.

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EVALUATION OF INTERFACE ROUGHNESS BY GRAZING INCIDENCE X-RAY DIFFRACTION

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AlAs/GaAs superlattice structures grown by molecular beam epitaxy were investigated by grazing incidence X-ray diffraction (GID). The surface orientation of the samples was near (001) with different values of miscut up to 3 degrees that produced different roughness of interfaces in superlattices. The triple axis diffractometer [1] at the beamline D4 of HASYLAB with a germanium (111) monochromator was used at a wavelength of 0.1336 nm. Rodscans of $220, \bar{2}\bar{2}0, 200$ and $\bar{2}00$ reflections for different values of the angle of incidence were measured.

For exit angles larger then the critical angle the curves show a large number of superstructure peaks. These correspond well to the layer periodicity of the multi quantum well structures. The experimental results are compared to the corresponding dynamical computer simulation including surface and interface roughness [2]. From the calculated curves corresponding best to the experimental ones an interface roughness up to 2.3 nm is determined. The roughness values obtained depend on the orientation of the diffracting planes to the direction of the maximal miscut.

The results obtained are compared to the results of topography, high resolution diffractometry, reflectivity measurements and measurements of diffuse scattering on the same samples.

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CURVABLE COLLIMATOR TOPOGRAPHY USING THE SYNCHROTRON SOURCE

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The principle of the double crystal technique with curvable monochromator-collimator (MC) crystal [1,2] is applied using synchrotron radiation. In this technique the curvature of the MC crystal is adapted to the sample curvature during the experiment obtaining large area double crystal topographs (compare [3] for flat MC crystal). This technique is well suited for the investigation of homogeneously bent samples like heteroepitaxial layer systems, especially when it is applied in reflection geometry. It is shown to have the full sensitivity of the double crystal technique for topography allowing for very short exposure times. Topographs obtained using adapted MC crystal curvature are compared with those using a plane crystal. The suitably curved MC crystal reflects the wave corresponding to the local Bragg condition in the sample by selecting appropriate wavelengths resulting in the increase of the sample area investigated.

As an example for a typical heteroepitaxial system a distributed Bragg reflector (DBR) structure of twenty pairs of AlAs (81nm) and GaAs (65nm) on a GaAs substrate is investigated (compare [4,5]). The diffractometer curve of this structure has a large number of superstructure diffraction peaks. The defect contrasts in satellite reflection topographs differ from those in the corresponding substrate reflection topograph.

The effects of additional monochromatization by a silicon double crystal monochromator are studied. The level of radiation background is reduced considerably. Topographs of the same area of the sample are compared with and without additional monochromatization. While the contrasts of the defects stay more or less the same, it is more difficult to obtain homogeneous large area topographs with additional monochromatization. The limits of the radius of MC crystal curvature (5m concave, 8m convex) are estimated.

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A HRD INTERACE STUDY IN THE WSi/Si AND OBLIQUELY DEPOSITED W/Si MULTILAYERS

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W/Si multilayers (MLs) are used as artificial X-UV mirrors. The interfaces play a decisive role to achieve the maximum reflectivity. The oblique deposition is a possibility to get smoother interfaces due to the increase of sideway motion and surface migration of the deposited atoms. The resistance against the interdiffusion and mixing at the interfaces can be increased by deposition of a WSi mixture instead of pure W. This can lead to an enhanced thermal stability and to smoother interfaces in the as-deposited state.

We applied the X-ray reflectivity and diffuse scattering measurements to study these possibilities. The measurements were done with $CuK_{\alpha 1}$ radiation on the STOE high-resolution diffractometer with the two-crystal +/- GaAs monochromator working at 400 reflection and cut under 3° with respect to the (100) plane. The samples were prepared by e-beam evaporation onto the Si (100) wafers. The angles α=0°, 38°, and 47° with respect to the surface normal (labelled as A,B,C) were used for the oblique deposition. The sample A was prepared as 10x(2.13nmW/12.77nmSi) starting with W and the ML periods Λ of B, C were reduced according to cosα as deduced from the simulation of the specular scans by the Fresnel optical computational code. The rms values of the interface roughness σ supposing the Gaussian distribution were 0.95nm, 1nm, 1.1nm for A,B,C, respectively. The intensity distribution in the reciprocal space was traced both by the ω-scans (the detector fixed) and 2Θ-ones (the sample fixed). The streaks around the Bragg points due to the correlated roughness were found for A and partially for B while they are almost absent for C. The scans were simulated within the DWBA approximation [1] taking the Λ and σ values from the specular simulations. Supposing the Gaussian height-height correlation function, i.e. 2D fractal character of the interfaces, the lateral correlation lengths ξ=24nm and 2.5nm were found for A considering the maximum correlation and for C considering the full loss of the correlation, respectively. The intermediate case B could not be simulated by the Spiller's model of the partially correlated roughness, where ξ increases with decreasing roughness correlation, which obviously is not our case.

The MLs $(W_{1-x}Si_x/Si)10x$ with $d_{WSi}/d_{Si}\equiv 0.55$ and x=0 (Λ=8.3nm), x=0.375 (Λ =7.6nm), and x=0.67 (Λ=8.7nm) were prepared starting with WSi and analyzed as described above. There was no change of σ=0.6nm with x. Contrarily, ξ=120nm for x=0 decreased to ξ=80nm for x=0.375 supposing the uncorrelated case. This tendency seemed to continue for x=0.67, however, neither both extreme cases of the roughness correlation nor the Spiller's model gave satisfactory simulation results.

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THE DIFFRACTION LINE PROFILES OF MESOPHASES. SMECTIC AND HEXATIC ORDERING

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There exists a wide range of intermediate phases (mesophases) between liquid and crystalline states, with order varying from that of an anisotropic liquid to that of an orientationally disordered crystal. There exists also an intermediate phase between the two-dimensional liquid and the two dimensional solid -- the hexatic phase, which possesses discrete symmetry with respect to rotations and reflections, like a crystal, but continuous translational symmetry. The peaks in the diffraction patterns of mesophases differ both from the delta-functions of crystals and the broad peaks due to short-range order of liquids. The shape of mesomorphous diffraction peaks is an essential feature providing information on their ordering.

Smectic order is one-dimensional translational order in a three-dimensional system. It is destroyed by thermal fluctuations of long wavelength and cannot exist in an infinite sample. In a finite sample, the strong thermal fluctuations cause intense x-ray scattering. Peak profiles of power law form, with a temperature-dependent index, were predicted theoretically and observed experimentally. However, the peaks are observed to be asymmetric, with the index considerably different from the predictions for an ideal smectic. We have shown that the results of various experiments on monomeric, polymeric, and lyotropic smectics can be explained naturally in terms of the real structure of the smectic, in which the mosaicity of the sample, misorientation and the finite sizes of the blocks are taken into consideration. An angular misorientation of only a few degrees of arc, which is quite small for a liquid crystal, has a considerable influence on the apparent index. A universal relationship between mosaicity and the index is found. The asymmetry of the diffraction peaks can also be explained as an average over misoriented blocks of the sample. Its temperature dependence is derived. The broadening of the x-ray peak due to distributed dislocations is described.

The structure factor is calculated for the original model of the hexatic phase by Nelson and Halperin, in which a two-dimensional crystal contains a gas of interacting dislocations. It is shown that the diffraction peaks are anisotropic Gaussians. The ratio of the longitudinal to the transverse peak widths depends on the sample size and decreases considerably when the dislocations interact. X-ray and electron diffraction experiments are explained without any phenomenological parameter.

The profiles of the diffraction peaks of mesophases with layer (smectic) and in-layer (hexatic) ordering are thus explained in terms of dislocations, their interaction, size and misorientation of the domains. The calculated peak profiles explain a wide range of various diffraction experiments.

DIFFRACTOMETRIC STUDIES OF ANOMALOUS TRANSMISSION OF X-RAYS IN SIX-BEAM LAUE DIFFRACTION

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An anomalous transmission of X-rays through a thick absorbing crystal is one of the fundamental phenomena of X-ray dynamical diffraction. The physical reason for this effect is the excitation of the standing-wave fields with intensity close to zero at the reflection planes. The structure of the standing-wave fields under conditions of multiple diffraction offers new possibilities for further reduction of interaction of X-rays with atoms and enhancement of anomalous transmission.

The most interesting configuration is the six-beam Laue diffraction case in which the minimum absorption coefficient is limited only by the Compton scattering. So far this effect have been studied experimentally mainly by the topographic methods. However, optical effects of X-ray spherical wave diffraction make observation and measuring of anomalous transmission nearly impossible.

The main experimental problem of direct diffractometric measurements of six-beam anomalous transmission is a high angular collimation of incident X-ray beam both in vertical and horizontal directions. In our experiments performed at the Photon Factory we used a new method of collimation based on the six-beam anomalous-transmission phenomenon itself. The enhancement of the anomalous transmission effect (in 3.3 times for $\mu t=12$) in comparison with the (220) two-beam diffraction has been observed for the first time in a direct diffractometric measurements.

Besides the fundamental interest the six-beam anomalous transmission may have important applications in X-ray optics. The six-beam collimator can be successfully used for two-dimensional angular collimation of X-ray beams in different diffraction experiments (three-beam multiple scattering, X-ray standing waves, total external reflection and others).

HIGH RESOLUTION X-RAY DIFFRACTION ANALYSIS OF ELASTIC LATTICE DISTORTIONS ASSOCIATED WITH SURFACE RIPPLING IN SiGe EPILAYERS

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Under certain conditions the surface of SiGe strained epilayers adopts a rippled morphology which is accompanied by an elastic deformation in addition to that of a smooth tetragonally distorted epilayer. We have applied diffraction space mapping to measure the nature and magnitude of these lattice distortions in a series of $Si_{1-x}Ge_x$ strained layers grown on Si by low pressure VPE. We show that the elastic distortions accompanying the periodic rippled surface morphology lower the elastic energy of the epilayer in accordance with theoretical modelling[1].

Atomic Force Microscopy and Transmission Electron Microscopy of the rippled SiGe surfaces reveal the existence of two sets of ripples each lying in one of the two <100> directions in the (001) epilayer surface[2,3]. A mosaic pattern of these ripples is observed on the epilayer surface. The symmetry of the ripple pattern allows X-Ray diffraction measurements to be performed in order to measure the specific elastic distortions associated with the ripple structure. In addition triple axis diffractometry permits the elastic distortions to be resolved into their lattice dilation and lattice tilt components. Recent measurements are presented which demonstrate that there is broad agreement between TEM measurements of the elastic distortions of {100} lattice planes normal to the epilayer surface and those of the (004) lattice planes measured using diffraction space mapping.

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DEFECT STRUCTURE INVESTIGATION OF SLIGHTLY IMPERFECT Si CRYSTALS BY MEANS OF X-RAY-ACOUSTIC METHOD

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The X-ray-acoustic method for determining of the structure perfection integral characteristics is suggested for slightly imperfect dislocation-free Si crystals with localized (micro) and distributed (macro) distortions.The method is based on the analysis of dependence of the distance Δx between two minima, arising in the spatial intensity profile I(x) of the X-ray beam diffracted by acoustically excited crystal [1,2], upon ultrasound frequency ν_S as well as of it's integrated reflectivity R_i . Using the data $\Delta x(\nu_S)$ for two selected reflections we have calculated the extinction lengths Λ (Table),

Sample	Si Fz		Si Cz	
Reflection	220	440	220	440
Λ, μm	36.54	54.27	36.5	53.62

which enabled us to identify the predominate type of structure disturbances for test crystals as well as to estimate the static Debye-Waller factors e^{-L} for sample with microdistortions. These data also made it possible to estimate L and the period of the main macrodeformation λ_m for sample containing simultaneously microdefects and periodic macrodistortions. Further analysis of the R_i values in framework of the dynamical theory developed for homogeneously distributed defects [3] provided us possibility for determination of the characteristic size r and concentration n of microdefects [4].This technique is shown to improve the obtained information quality and diffraction data reliability. Such approach has been used for studying the structure perfection of Czochralski-grown (Cz) (L_{220} $=1{,}9*10^{-3}$, L_{440} $=7{,}6*10^{-3}$, λ_m $=200$ μm) and Float-zone (Fz) (L_{220} $=1{,}1*10^{-3}$, L_{440} $=4{,}3*10^{-3}$, r=1,. μm, $n=2{,}9*10^{11}$ m^{-3}) silicon crystals.

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THE DISTRIBUTION OF STRAIN AND TILT IN GRADED BUFFER LAYERS

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In buffer layer technology, in addition to measurement of layer composition and relaxation, it is necessary to find a method for quantification of the crystalline quality of the relaxed buffer layers. The technique of high resolution reciprocal space mapping is ideal for this purpose, as it can reveal details of the layer unit cell dimensions and the distributions of tilts and mosaic spread. We investigate the relaxation behaviour of two graded layer structures comprising InGaAs layers grown by MBE on (001) oriented GaAs substrates. Both structures have a linear graded buffer layer of $In_xGa_{(1-x)}As$ with x increasing from 0 to 0.3 over 1μm. One of the samples has a capping layer with thickness 300nm and composition x=0.21. By studying the shape and distribution of the diffuse scatter around the layer Bragg peaks for the 004 and 115 Bragg reflections we are able to quantify the range of lattice parameters and tilts in the layers. Figure 1 below shows a typical reciprocal space map around the 004 Bragg peak for the capped sample, illustrating the measurable parameters.

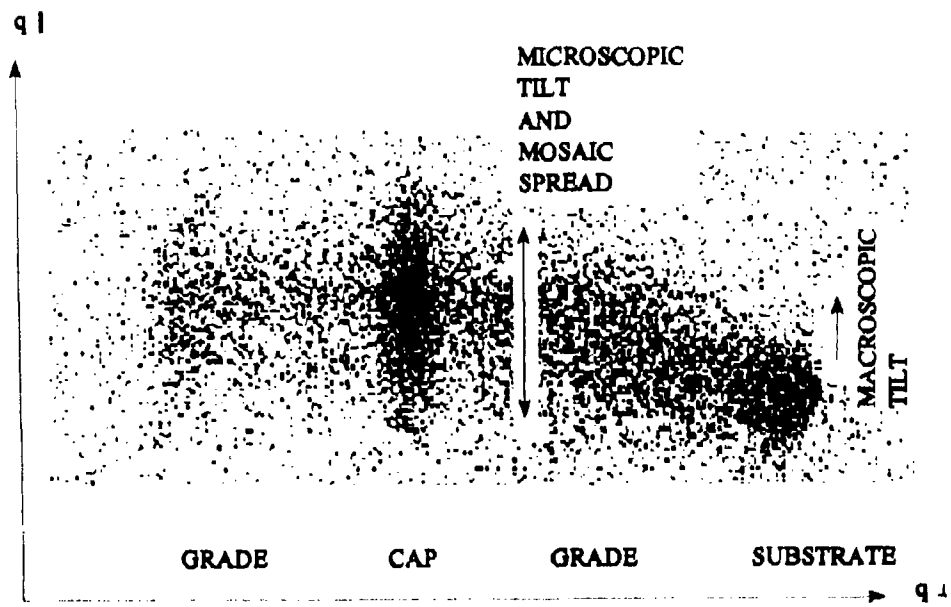

Fig 1 Reciprocal Space Map with grey scale indicating log (intensity) for the 004 Bragg reflection from a 1 μm thick InGaAs graded buffer layer with an InGaAs constant composition capping layer, grown by MBE on an (001) oriented GaAs substrate.

DOUBLE-PLANE COLLIMATION OF X-RAYS FOR HIGH-RESOLUTION EXPERIMENTS

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The majority of collimating systems in X-ray optics provide precise angular collimation of X-rays in one plane only. This collimation produced by Bragg reflections from single crystals can be as fine as $\delta\Theta \simeq 10^{-1} - 10^{-2}$ seconds of arc. The angular divergence of X-rays in the other plane is usually formed by slits, pinholes or X-ray mirrors and is not better in most cases than $\simeq 10^{2} - 10^{3}$ seconds of arc. That suits well for most experiments. However, some new X-ray diffraction geometries extensively used in recent years (grazing incidence diffraction, multiple diffraction, extremely asymmetric diffraction, etc.) require the incident beam be collimated in both mutually perpendicular planes with an accuracy not worse than $\simeq 1$ second of arc. Moreover, the high monochromatization of X-rays must be provided as well because the dispersion is usually not compensated in these geometries.

In the present report, three possible ways of X-ray double-plane collimation are discussed:

1. With 3 successive Bragg monochromators arranged in two planes [1],
2. With 6-beam Borrmann effect [2,3],
3. With Renninger effect [4,5].

The collimation based on the Renninger effect is shown to be most advantageous. The experimental study of the double-plane collimation produced by the Bragg-case Renninger effect is carried out. The data are compared with theoretical calculations based on three-wave dynamical diffraction theory. The analysis includes also a detailed consideration of dispersion effects.

Then, the effects of asymmetry and of different combinations of primary and secondary planes on double-plane collimation parameters are studied by means of computer simulations. It is shown that the most effective collimator can be based on the Laue case Renninger effect. Such a collimator is able to combine the advantages of a Bragg case Renninger collimator (using of 3-beam diffraction only) and a 6-beam Borrmann collimator (the absence of fine structure and long tails in the produced X-ray beam).

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Deposition Controlled Surface Roughness of Thin Gold Films Studied by X-Ray Reflectivity

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Non-destructive surface and interface characterization is of great interest both in applied and basic research. It is well known that even interfacial regions extending over a few monolayers can severely degrade device performance [1]; with further miniaturization the need to control and monitor interface quality in deposited layers will still increase. As far as basic research is concerned, the growth of thin films recently has received much attention from theorists [2] and experimentalists [3] due to the predicted scaling behaviour of self-affine surfaces and the associated roughness exponents.
For both purposes specular and diffuse x-ray reflectivity under grazing angles was proved to be a sensitive tool to investigate statistical interface properties like RMS-roughness in growth direction and lateral correlations [4,5].
We have applied this method to characterize the surface of thin gold films deposited on silicon substrates. Experimental data were taken with synchrotron radiation at HASYLAB/DESY on samples grown under identical conditions but to different film thicknesses between 20 and 500 nm. A second series of samples with identical film thickness was investigated to study the influence of growth rates from 0.03 to 1 nm/s on surface roughness
The data have been evaluated by simulation assuming a lateral correlation function of the type $C(r) = \sigma^2 \exp(-(r/\xi)^{2h})$ The results are compared to predictions for kinetic roughening

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ANALYSIS OF SURFACES AND LAYERED STRUCTURES BY X-RAY SCATTERING

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A wide variety of x-ray methods are used for investigating surfaces and thin layers.

Angular dependences of the specular reflectivity provide reliable data about material densities and layer thicknesses. The root-mean-square (rms) roughness of surfaces and interfaces is also frequently deduced from reflectivity curves. However, detailed scattering theory indicates that such results can be considerably distorted. Measurement of the nonspecular scattering yields better information about rough interfaces (including lateral characteristics and vertical correlation of individual interface profiles). On the other hand, this approach requires more sophisticated experimental equipment and theoretical interpretation.

Recently, new theories of the diffuse x-ray scattering from surfaces[1] and layered systems[2,3] based on the distorted-wave Born approximation have been developed. By using these models, a more complex characterization of non-ideal layered systems is possible. The use of nonspecular scattering to study surfaces, thin films, and multilayer structures is demonstrated for several types of samples namely glass, fused silica, single Al layer and high-quality Fe/C multilayer with rms roughness less than 0.5 nm. The experiment has been carried out using a two-axis reflectometer and CuK_{α} radiation produced by a 18-kW x-ray generator with rotating anode.

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X-RAY MULTIPLE SUCCESSIVE DIFFRACTION AND BEAM TRACING

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In X-ray topography and high resolution diffractometry, single and multiple crystal coplanar arrangements are used depending on the required sensitivity, resolution, intensity, and other parameters. Special beam conditioners consisting usually of two adjustable grooved crystal blocks [1] are applied to form the X-ray beam by means of coplanar multiple successive diffraction. Contrarily, non-coplanar successive asymmetric diffractions (diffractors) have been successfully used to obtain X-ray magnified image [2].

Multiple diffraction is usually studied because of its dynamical effects in multiple simultaneous diffraction [3]. Conditions for coplanar and non-coplanar multiple successive diffraction (based on geometrical approach and Bragg equation in vector form) and basic imaging equations for the beams to pass have bee.. given in [4]. Using the equations, a monolithic X-ray magnifier integrating two non-coplanar asymmetric diffractors has been devised and constructed [4]. Similarly, the equations have been used to perform beam tracing and Monte Carlo simulation of X-ray topographs in single and double crystal (flat and curved crystals) symmetrical and asymmetrical diffraction with the aim to study the effect of geometrical parameters on the X-ray topographs.

Our further study of the multiple successive diffraction includes two-beam dynamical approach to the individual diffractions. The procedure thus takes into account the effect of refraction and asymmetrical diffractions on the rocking curve position. The effects of multiple dynamical diffraction have not been considered which means that no information on the dynamical gain or loss of intensity [3,5] can be obtained from the computations. About 20 millions of combinations of three crystallographic planes for Si, Ge, and GaAs crystals, and for the most common X-ray wavelengths have been tested with the aim to find arrangements suitable for monolithic non-coplanar, near coplanar, and coplanar devices. In addition to a matching parameter for the diffraction cones of the diffractin vectors considered, a coplanarity parameter for the incoming and successively diffracted beams has been evaluated in the rules of choice. In addition, combinations of lower order diffractions and Bragg angles relatively far from 45 degrees have been given preference in order to preserve as much intensity as possible in the non-coplanar case.

Based on the computations, a monolithic X-ray magnifier integrating a monochromator and two asymmetric non-coplanar diffractors has been devised and constructed. Similarly, a monolithic beam conditioner close to the (-n,n,m,-m) type, with relatively high coplanarity parameter, has been realized and tested. Its important advantage is that the incoming and the outcoming beams are nearly parallel.

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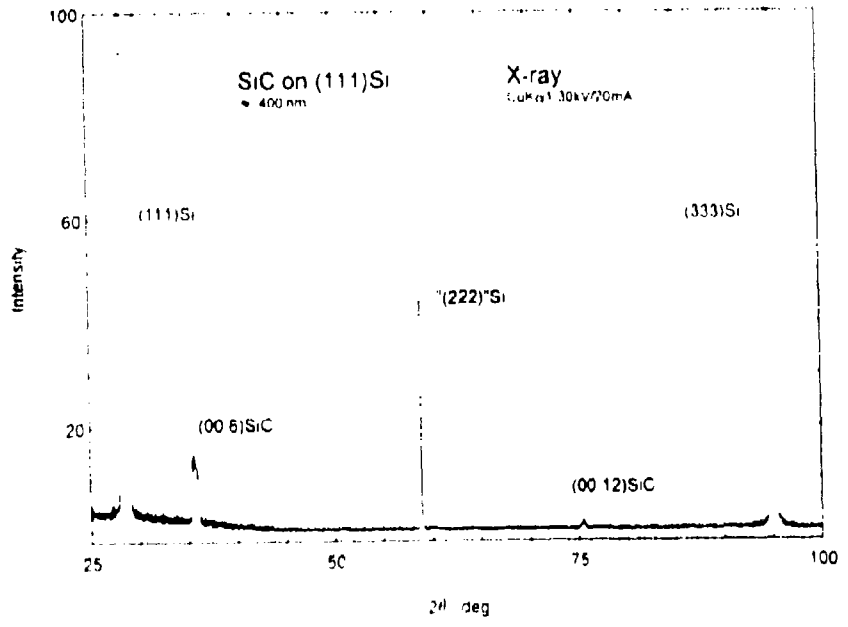
Thin SiC films on (111)Si wafers

Thin SiC films with a thickness of 100 - 500nm have been produced on (111) oriented Si wafers using solid state evaporation. Growth rates of 0.2 - 0.6 µm/h have been achieved at 750° - 900° C

The X-ray diffraction pattern obtained from a 400nm SiC film (see Figure) exhibits two typical peaks due to the reflections at the (00.6)-plane of α-SiC or (111)-plane of β-SiC. No other SiC peaks were observed. This implies that the film crystallites have a preferred growth orientation nearly perpendicular to the plane of the close-packed structures. The measured orientation distribution function was characterized to be Gaussian with FWHM of 2.5°

Currently we are getting additional informations concerning properties and behaviour of these SiC thin films by using atomic force microscopy and IR-spectroscopy



The work was supported by the Sonderforschungsbereich 196 (projects A03 and C01)

STUDY OF DEFECTS AND ULTRASOUND WAVES IN NEARLY PERFECT HTSC CRYSTALS OF $Nd_{2-x}Ce_xCuO_{4-\delta}$ BY NEUTRON AND X-RAY TOPOGRAPHY

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Methods of neutron and X-ray diffraction and topography have been developed for study of defects and acoustic properties of HTSC crystals.

It was established that some nonsuperconducting $Nd_{2-x}Ce_xCuO_4$ ($x=0-0,17$) [1-3] and superconducting $Nd_{1.85}Ce_{0.15}CuO_{4-y}$ crystals have a very high perfection. As a result, the dynamical effects: narrow rocking curves (close to theoretical ones for ideal crystals), Borrmann effect and Pendellosung oscillations were observed in neutron and X-ray scattering. The defects (dislocations, individual small angle boundaries, elastic stresses, concentration inhomogeneity) were revealed in the crystals. The dislocation type and density and Burgers vector orientation were determined.

The influence of ultrasound waves on X-ray diffraction in perfect $Nd_{2-x}Ce_xCuO_4$ crystals (at Borrmann effect condition) was investigated. It was demonstrated that ultrasound waves may considerably decrease the intensity. The systems of periodic gaps were found on frequency dependence of X-ray intensity and connection of the gaps with stationary acoustic waves was established. The stationary acoustic field was visualized by X-ray topography. The acoustic wave amplitude and polarization and the velocity of longitudinal and transverse sound were determined from diffraction data.

It was found that the influence of ultrasound waves on diffraction intensity at Borrmann effect condition increases when the sound wavelength approaches to extinction length. It was shown that the effect is connected with the resonance interaction of X-ray and acoustic fields and the Borrmann effect suppression (X-ray acoustic resonance).

Possibilities of application of topography and diffraction methods for investigation of the superconductivity are discussed.

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DETERMINATION OF STRAINED SUPERLATTICE STRUCTURAL PARAMETERS.

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X-ray diffractometry is widely used for a superlattice (SL) structural parameters determination, when SL are of high quality and match to substrates. This communication deals with strained SL containing the defects inside the layers. Double and triple axis settings were utilized. The InGaAs-GaAs SL on GaAs substrates as well as AlSb-GaSb SL in GaSb/GaAs structures were studied.

Similar to nearly perfect SL, the period and the average deformation were obtained from the satellite positions in the double crystal rocking curve. We used the asymmetric Bragg diffraction to extract the in-plain data, which allowed us to judge about the presence of misfit dislocation (MD) networks. As for the individual layer thicknesses and strain, they are known to be determined by using a simulation of the curves. Since the basic principles of the simulation in this case still remain unclear, we, considering no a general approach, attempted the simulation according to the following. One point is that no the relative intensities, but the integral reflectivities are compared. Second, there is an approximation of the lattice tilt by the Gaussian distribution. After a fitting of the integral reflectivites is reached, one finds the deformation components as well as the thicknesses of SL layers. The use of tilt approximation permits the simulation of the peak shapes.

In order to assess the quality of AlSb-GaSb SL layers we applied to them the triple axis scans. It was concluded that the peak broadenings in the transverse to the reciprocal lattice vector H direction and those in the parallel were unvariable between the satellites. We interpreted the both as being due to a high density of threading dislocations. Examining InGaAs-GaAs SL, we detected a presence of MD networks in the SL-substrate interfaces and found that the SL layer planes sloped from the substrate ones.

All of the extracted parameters were confirmed by direct TEM observations.

FLUX AND TEMPERATURE DEPENDENCE OF LATTICE EXPANSION IN LT GAAS THIN FILMS.

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GaAs layers grown at reduced temperatures (180 ^{o}C < T_{gr} < 300 ^{o}C), the so-called LT GaAs, have an increased lattice constant in the direction of growth which is due to excess of As [1,2].We have shown that this lattice expansion and hence As excess depends also on the growth flux ratio $R = J_{As4}/J_{Ga}$ [3]

In order to understand the mechanism responsible for this effect we have studied the lattice expansion dependence in LT GaAs thin films on growth temperature for different flux ratios using High Resolution XRD.TEM has been used for the study of As precipitates in the samples after annealing at 600 ^{o}C and the density and diameter of As precipitates will be discussed in connection with the lattice expansion. Figure 1 shows the relaxed lattice constant increase of LT GaAs layers, grown by MBE technique, on flux ratio R for different temperatures. $(\Delta a/a)_r$ increases linearly with respect to R and reaches a saturation value which depends on temperature. Samples grown by Atomic Layer Epitaxy have lower saturation values of lattice expansion.

The saturation in $(\Delta a/a)_r$ indicates that there is a saturation of adsorption of excess As in the GaAs lattice which depends on growth temperature. The dependence of $(\Delta a/a)_r$ on flux ratio and on temperature will be discussed on the basis of a model of As incorporation during growth at low temperature.

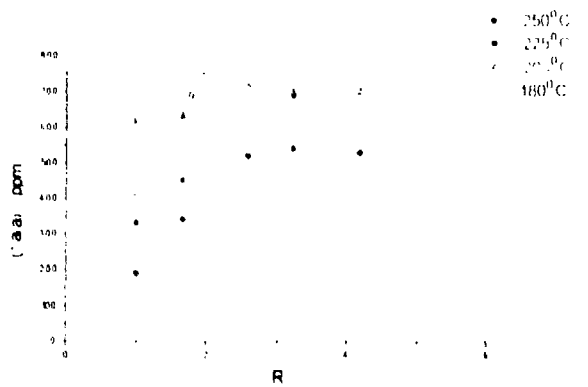

Figure 1 . Relaxed lattice expansion $(\Delta a/a)_r$ of GaAs at different flux ratios $R = J_{As4}/J_{Ga}$ and different growth temperatures.

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HIGH RESOLUTION XRD STUDY OF $Al_xGa_{1-x}As$ LAYERS GROWN AT REDUCED TEMPERATURE.

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$Al_xGa_{1-x}As$ layers grown by MBE at 200°C-300°C contain excess As as much as 1% [1]. After annealing at 600 °C they become of high resistivity[2] ($>10^8$ Ωcm) and they offer good isolation in MESFET's devices. Excess As creates precipitates with density and size depending on growth temperature and annealing procedure. High crystalline quality and smooth surface in LT layers is needed in order to achieve high electron mobility and transconductance in device applications[3].

In this work we investigate the crystalline quality, lattice expansion and surface morphology of $Al_{0.28}Ga_{0.72}As$ grown by MBE at 200°C- 300°C by High Resolution XRD and SEM. (1x1) Rheed pattern was observed during growth of 1μm LT AlGaAs. The surface is smooth for $T_{gr} > 250$ °C and rough for $T_{gr} < 250$ °C. The surface roughness is due to decrease in surface mobility and diffusion length of group III atoms[4].

Lattice expansion due to LT growth was determined from the difference in the peak splitting $\Delta\theta$ of the (004) symmetric Bragg reflection between as-grown and annealed LT AlGaAs. Coherent growth of the layers was assumed after recording the asymmetric (115) rocking curve for low and high angle of incidence. The lattice expansion decreases as T_{gr} is increasing due to decrease in excess As concetration.The dependence of the relaxed lattice expansion $(\Delta a/a)_r$ on growth temperature will be discussed. We have observed a much lower lattice expansion in LT AlGaAs layers compared to that of LTGaAs layers grown with identical conditions.The crystalline quality of LT AlGaAs epilayers is very good as it can be seen from the presence of thickness fringes in the (004) Bragg reflection rocking curve.

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HIGH-RESOLUTION DIFFRACTION AND X-RAY STANDING WAVE STUDY OF Si/Ge SUPERLATTICES

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Si/Ge superlattices (SL) have recently attracted much interest because of their optoelectronic properties, which include the possibility to obtain direct optical transitions (1) and the vast field of intersubband transitions (2). These properties could open the way to integration of advanced optoelectronic functions with the well developed Si technology. A key point in order to obtain the required characteristics is the structural perfection of the SL, such as the thickness uniformity of the individual layers, the minimization of extended defects and the crystalline quality and abruptness of the interfaces. X-ray diffraction and X-ray specular reflection can give accurate structural information about the SL, but as usual only the modulus of the structure factor can be directly determined with these methods. A direct determination of the phase can be obtained by applying the X-ray Standing Wave (XSW) technique to these structures. In XSW measurements from very thin layers the fluorescence from the overlayer atoms is generally measured in correspondence with the substrate diffraction peak. However, in the case of SuperLattices, the XSW field generated in the substrate goes rapidly out of phase with respect to the SL periodicity. We made therefore experiments and calculations with the XSW field generated in the SL itself, measuring the Ge fluorescence yield as a function of diffracting angle in correspondence with the satellite peak of the (004) diffracting planes. From the combined information of the diffracted intensity and of the fluorescence yield, detailed information about the SL structure are derived.

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ABSOLUTE MEASUREMENT OF THE LATTICE PARAMETER OF PERFECT DIAMONDS BY THE DIVERGENT-BEAM (PSEUDO-KOSSEL) X-RAY METHOD

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A modern embodiment of the classic work of Lonsdale (1947) has been developed and applied to natural and synthetic diamonds that have been fully characterised by modern methods such as X-ray and cathodoluminescence topography, and Fourier Transform infrared absorption spectroscopy. The specimen volumes studied are dislocation-free, with damage-free polished surfaces, and have been demonstrated to possess high homogeneity of impurity content together with high long-range lattice perfection. The integrated intensity of Bragg reflections from such perfect diamonds is probably at least 100 times less than from those diamonds studied by Mrs. Lonsdale. Consequently, special techniques have had to be introduced to cope with such weak patterns. Following her method, we study transmitted divergent beam patterns in the vicinity of certain Kossel cone intersections where the pattern is very sensitive to small variations in the ratio λ/a, λ being the CuKα_1 wavelength, and a the diamond lattice parameter (Kossel 1936).

In our experiments the specimen is placed in the specimen chamber of a scanning electron microscope (SEM). This instrument has proved well suited for generating divergent-beam X-ray diffraction patterns (Tixier and Waché 1970, Dingley 1978). Characteristic radiation of a suitable element is produced by electron bombardment of a thin film of that element in contact with the specimen crystal. The resulting diffraction patterns are known as 'pseudo-Kossel' patterns, but their geometry is identical with those observed in the original experiments of Kossel & Voges (1935) in which the specimen itself (e.g. a copper single crystal) formed the target of an X-ray tube. In our experiments a 2 μm-thick copper film is evaporated on to the diamond crystal to act as a source of CuKα X-rays. Features of our experimental technique include a) strict temperature control of the specimen by means of a miniature Peltier heating/cooling device, (b) a high-dispersion camera providing an evacuated path of 700 mm between specimen and photograph film or plates, (c) use of modern X-ray recording emulsions that combine fine grain size with high X-ray absorption efficiency, (d) introduction of a 'film-stacking' technique for building up the signal-to-background ratio, and (e) digital scanning photometry.

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STUDY OF DEFECTS GENERATED IN CZ SI DURING TWO-STEP ANNEALING

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The aim of the study was to detect and characterize defects generated in Czochralski-grown silicon single crystals during two-step annealing processes: at 1173 K for 1h15m and at 1423 K for 4 hrs, 6 hrs, 8 hrs or 10 hrs, respectively. X-ray topography and diffractometry as well as transmission electron microscopy (TEM) were used for the investigations.

Lang topography revealed contrasts characteristic for spherically symmetrical defects. Besides defects with two- and six-fold symmetry were observed. Their density decreased with increasing annealing time. The size of their contrast was about 70 μm and it tended to increase with the anneal time. For a sample annealed at 1423 K for 10 hrs also curvilinear dislocations, ending on microdefects, were detected.

Investigations of the overall perfectness of the crystal lattice in regions of small microdefect density (or of their absence) revealed a distinct asymmetry of the strain distribution. On section topographs recorded with the symmetrical 220, 440, 660 and 880 reflections a clear pattern of the Kato fringes characteristic for perfect crystals was observed, while for the asymmetrical 333, 444, 551, 771 and 531 reflections the strain gradient was so strong that no fringes were visible. The only exceptions where weak fringes were detectable was the 333 reflection for the sample with the shortest anneal time of 4 hrs as well as the sample where stresses had been relieved by the generation of dislocation lines. Preliminary high-resolution diffractometric investigations of these samples revealed no important diffuse scattering for symmetrical reflections.

TEM investigations revealed the presence of plane defects in the form of dislocation loops of a very small density and small size (0.25-1 μm). For the sample annealed at 1423 K for 4 hrs no defects were observed while in the sample annealed for 6 hrs a significant number of larger defects (1 μm) was found. For the longest annealed (10 hrs) sample where strains were relieved by dislocations defects are very scarce and have small size (< 0.25 μm).

THERMAL EXPANSION AND COMPRESSIBILITY OF GALLIUM NITRIDE

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Properties of gallium nitride are much less known than of other III-V semiconductors (GaAs, InP, etc.). This situation originates from a difficulty to grow single crystals because of a high equilibrium pressure of nitrogen. However, recently gallium nitride attracts a lot of interest, because its wide band gap (3.4 eV) makes it to be a main candidate for a construction of a blue laser, other optoelectronic devices, as well as for high temperature electronics.

In this work we present results of measurements on gallium nitride performed by using an X-ray diffractometry (Bond method, double crystal, high resolution) at a wide range of temperature (77-770 K) and pressures (1-8000 atm). The samples were in the form of unique single crystals grown at pressures of about 12000 atm and temperatures of about 1300 K and in the form of epitaxial layers grown on sapphire.

The following physical properties of GaN have been evaluated

a) real structure of bulk crystals

b) concentration of defects in the layer induced by the lattice mismatch with the substrate

c) thermal expansion and how it is influenced by presence of the substrate which has a higher thermal expansion coefficient

d) compressibility

The results of the structural examinations are correlated with the measurements of electrical and optical properties of the samples.

Apart that, the work illustrates well the usefulness of X-ray diffractometry combined with changeable temperature and pressure conditions.

DISLOCATION EMISSION UNDER MIXED MODE LOADING IN SILICON

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Stress relaxation at crack tip can be achieved through dislocation emission. Because of the inhomogeneity of the stress field, the choice of the Burgers vectors of the actived slip systems cannot be simply determined by a Schmid factor. Some attempts have been made to put forward a criterion based on averaged values of the resolved shear stress acting on the dislocation [1]. However, being not always fulfilled, an other one, based on the crack shielding concept has been proposed [2]. It needs to be checked on a wide variety of orientations.

In order to bypass the geometrical limitations imposed by the few available crystallographic orientations (two possible orientations of the Thomson tetrahedron with respect to a {111} cleavage planes, an only one orientation for easy cleavage along {110} planes) mixed mode loadings are required. For the well suited thin tapered DCB samples used for such studies in silicon, it was generally easier to develop mode I + II loadings so far. However this paper will present results obtained on I + III loading due to a warp of the crack during its low temperature propagation.

The pre-cracked samples are loaded at a temperature high enough for silicon to be ductile. The number of active sources increasing rapidly with the stress level, a two steps loading is used in order to develop diluted plastic zones : the high load applied for a minute in order to activate a few sources is decreased to a low value in order to expand the configuration of already emitted dislocation without further germination.

This paper will compare a plastic zone, as observed under pure mode I loading to an other one developed under modes I + III loadings. The discussion of the activated slip systems, as characterized by X-Ray topography, will emphasise the relative contribution of the two modes, through the stress intensity factors K_I and K_{III}.

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X-RAY DIFFRACTION STUDIES OF THE POROUS SILICON THIN LAYERS

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The recent discovery of photoluminescence and electroluminescence properties in porous silicon (PS) has produced a great interest. In general, these properties are attributed to quantum confinement effects in the nanometric size crystalline in PS. To test the quantum confinement hypothesis, it is necessary to determine the porous silicon parameters (size, shape, deformation, porosity ...). The PS parameters (size and morphological informations) were obtained by X ray diffuse, small angle and close to Bragg peaks scattering. However, high-resolution Bragg diffractometry has been used for the deformation evaluation only. The aim of the present work is to start a quantitative analysis of the PS layer diffraction peaks by using double and triple-crystal diffractometry measurements.

In order to have samples with a good crystalline properties, low porosity heavily doped p silicon samples ($1 \cdot 10^{-2}$ om·cm, (100) orientation) were used. PS layers (going from 20 to 0.03 μm) were produced by anodization in a mixture of hydrofluoric acid and of ethanol with a constant current of 10 ma/cm giving a porosity of 10%. For our high-resolution experiments, we used the RD Philips diffractometer. In the thicker samples, narrow Bragg peaks were observed for as grown samples, with profiles close to those of the dynamical diffraction theory. For thin samples (in the 0.1 μm range) the PS Bragg peak was not resolved, but appeared as a shoulder on the substrate peak; thickness fringes were observed, showing that the porous layer surfaces were well defined. For analysis of submicron layers diffraction curves, the kinematical approximation was used. It was shown that, for good simulation, PS layer must be divided on the main and intermediate sublayers. The first sublayer average deformation and Debye-Waller factor were found to be $5 \cdot 10^{-4}$ and 0.6 accordingly. The second sublayer thickness was nearly 20 nm and did not vary for the investigated samples. The intermediate layers parameters were between those of the main layer and the substrate.

X-RAY HIGH-RESOLUTION DIFFRACTOMETRY INVESTIGATION OF SULPHUR IMPLANTED AND PULSE LASER ANNEALED InSb (111) SUBSTRATES.

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The triple-crystal X-ray diffractometry (TCD) is an important technique for separate registration of the diffuse and dynamic components of the total scattering as well as precision measurements of reflection curves far away from the exact Bragg angle (truncation rods method). The semiconductor crystals structure investigations are of interest because of their influences on electro-physical properties of these materials. The pulse laser annealing (PLA) is outlook method for a subsurface layer structure restoring and an electrical activation of implanted impurities. It has many advantagies as compared with the common used thermal annealing. The influence of nanosecond and millisecond PLA on implanted crystal structure was studied by DCD and TCD methods, but now the silicon crystal PLA have been studied more exhaustive only. There is not at all some information about InSb monocrystall pulse laser annealing.

In the present paper we investigate the subsurface layer structure distortion of sulphur ion implanted (100 kev, D=10e+17at/cm^2) and pulsed laser annealed (ne tim laser, 10ns duration, Q=0.1-0.2 J/cm^2) InSb(111) crystal at 300K and 77K temperaturies by X-ray high-resolution techniques. It has been shown that this implantation leeds to full amorphous layers. After PLA 0.3mkm thickness liquid phase epitaxial layer with decreased (-1.6e-3) lattice spase and with low dislocation densities was is created at room temperature. This PLA at 77K is enough for solid phase epitaxial layer regrowth with low crystal perfection.

APPLICATION OF MULTIPLE GROOVE CHANNEL-CUT CRYSTALS IN HIGH-RESOLUTION X-RAY DIFFRACTION

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Both in the double and triple axis X-ray diffraction configurations, a major requirement is to switch between X-ray optical settings of different angular range and wavelength bandpass. This is generally achieved either by changing beam conditioner and analyser crystals or changing the reflections without replacing the crystals. We have adopted a new approach which exploits the facility to alter the angular acceptance and divergence of a channel cut crystal by changing the angle of cut with respect to the diffracting planes used. For such asymmetrically cut, parallel channels, the input and output divergences and beam widths remain matched.

In this paper we describe the performance of an X-ray optical element comprising a single crystal block of silicon containing two beam channels cut respectively parallel to and 17.65° from the (011) planes. The device is cut to such dimensions that with CuKα radiation the beam makes two reflections in the asymmetrically cut channel (asymmetry factor b = 2.5) and four reflections in the symmetrically cut channel. Parallel, lateral, translation of the two elements permits a very rapid switch between high intensity and high resolution settings.

We have used two of these elements, together with high precision slides, to construct a novel, duMond configuration monochromator. In the high intensity setting, the angular output divergence FWHM is 11.5 arc sec with a corresponding dispersion $\delta\lambda/\lambda = 1.4 \times 10^{-4}$. This is very similar to that for the Bartels device which uses the symmetric 022 reflection from germanium. When set on the peak of the $CuK\alpha_1$ line from a fine-focus X-ray tube running at 40kV 50mA, the device delivers 650,000 c.p.s. in a beam of dimension 14 x 0.75 mm² at the sample. Due to the complementary nature of the two asymmetric reflections, the spatial width of the exit beam is the same as that of the entrance beam. In the high resolution setting, reached by parallel lateral translation of the two elements, the angular FWHM is 4.4 arc sec and the dispersion 4.9×10^{-5}. The intensity, under equivalent conditions to that above, is 75,000 c.p.s at the sample.

The device has also been used as a variable acceptance analyser, which permits rapid switching in resolution during triple axis reciprocal space mapping. With minimal readjustment, a simple lateral translation of the analyser enables the experimenter to examine in detail specific features at high resolution (but often therefore with low recorded intensity), make a wider range map with a larger sampling volume in reciprocal space or perform an un-analysed double axis measurement. We present exemplary data.

TOPOGRAPHY IN COHERENT BEAMS OF A NEUTRON INTERFEROMETER

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High-resolution two-dimensional position sensitive detectors permit to investigate the spatial intensity distribution in coherent beams of thermal neutrons outgoing from a neutron interferometer. The detailed analysis of observed Moiré patterns of an empty interferometer can yield very useful information on dominant negative influences decreasing the observed interferometric contrast. Moreover, in the case of sufficiently wide neutron beams passing through the interferometer, the neutron "topographic" experiment can be performed when putting a sample into one of coherent beams between the interferometer slabs. The resulting topographs may exhibit a contrast being sensitive to the relative neutron phase shift $\Delta\Phi=\rho\lambda t$ (ρ - scattering length density, λ neutron wavelength, t - neutron path in the sample) introduced by passing one coherent beam through the sample. Thus, the phase topography gives information on local variations of both thickness and scattering length density of a tested sample. It may be of a great importance for current interferometric experiments determining precise values of neutron scattering lengths of nuclei. Some computer processed topographs of an edge shaped Ge waffer and an isotopic pure sample of ^{208}Pb will be presented. The results were obtained on interferometric facilities in NPI Řež and HMI Berlin.

TIME RESOLVED INVESTIGATIONS OF THIN ORGANIC FILMS BY MEANS OF ENERGY DISPERSIVE X-RAY REFLECTOMETRY

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The total thickness and the periodicity of lamellar films has been evaluated by measuring the X-ray reflectivity. For this purpose the angular dispersive technique is often applied. Another way to determine X-ray scattering spectra is the energy dispersive technique. In this case the angle of incidence remains constant and the reflected intensity is detected in dependence of the energy using a Si(Li)-detector and a multichannel analyzer. For quantitative evaluation the measured spectrum has to be corrected by the detector sensitivity and the energy dependent absorption of sample and air. This restricts the accuracy for measurements on the absolute scale. The advantage of the energy dispersive system is the possibility to use the whole X-ray spectrum. Due to the fixed angle of incidence the illuminated area does not change during the measurement. Periodically repeated measurements of about a few minutes are sufficient to get statistical reliability for time resolved processes like melting or rearrangement phenomena in organic multilayers.

The application of the energy dispersive method is demonstrated at two examples: the evaluation of phase transition kinetics in multilayers of fatty acid salts (Ref. [1]) and the investigation of photoinduced structural responses of thin organic films built up by an amphotropic azobenzenecopolymer (Ref. [2]). In both cases the have been prepared by the Langmuir-Blodgett technique. The scattering spectra have been recorded in several time intervals at fixed temperatures T.

For LB-films of fatty acid salts T has been fixed to $T = T_S - \Delta T$, where T_S is the melting temperature and ΔT has been set to 4 - 12 K. One can observe a time dependent disappearing of the mean structural peaks of the initial LB-phase. After about two hours new Bragg peaks appear which correspond to a lamellar spacing smaller than in the LB-phase. The time constant of the phase transition depends on the temperature and disappears at T_S.

Photochromic copolymers with rod like azobenzene units as side groups show interesting changes of the supramolecular structure on annealing and on irradiation with UV and visible light. Upon illumination in the UV range one can observe a reorganization of the initial LB- film into a new phase. The intensity of the respective Bragg peaks depends on the temperature and the wavelenght of irradiation. This can be interpreted by a lateral melting or growth of domains in the film. The activation energy of this process can be obtained by time and temperature dependent measurements of peak intensity.

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X-RAY REFRACTION INTROSCORY OF THE BIOLOGICAL OBJECTS

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Method of refraction X-ray introscopy was proposed by authors in [1,2]. Method is based on the use of the double perfect crystal setup with the object placed between the crystals and the image taken in the non refracted beam. In this case the structure of very low absorbing details could be observable as contrast for the conventional X-ray introscopy. In the present work this method was performed with the help of the channel-cut crystal with wide channel and the scanning technique which is used in topography. Images of model and real biological objects were obtained at the Mo Kα radiation and angular resolution about 1 second of arc. Both photo registration and computer controlled TV detector were used. The observed and calculated contrast were compared for the model objects with different size and refraction index for both types of registration. Feasibility of method with the use of multibeam diffraction, which is essential for SR, is analyzed. In this case simultaneous registration of several projections is possible. Possibility of the use of this method for hard radiation (about 30keV) is discussed.

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X-RAY DYNAMICAL DIFFRACTION IN PERFECT SUPERLATTICES IN CONVENTIONAL BRAGG AND GRAZING-INCIDENCE GEOMETRIES.

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A new approach for description of dynamical diffraction in ideal superlattices (SL) is considered. The proposed method differs from the existing ones and is based on fundamental Bloch theorem. In accordance with this theorem fundamental solutions of wave equation in periodic media for arbitrary value of z must satisfy the following equality:

$$D_{0,h}(z+T) = \rho D_{0,h}(z),$$

where $D_{0,h}(z)$ are the amplitudes of the refracted and diffracted waves, respectively; T is the period of SL and ρ is called multiplicator. In conventional geometry the Bloch theorem is applied to the solutions of Takagi-Taupin set of differential equations and the whole problem of diffraction in SL reduces to determination of eigenvalues and relevant eigenvectors of a 2 x 2 matrix /1/. In grazing incidence geometry the reduced set of Maxwell equations is used and all operations are performed with 4 x 4 matrices /2/.

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DYNAMICAL EFFECTS IN MAGNETIC X-RAY DIFFRACTION ON SPIRAL MAGNETIC STRUCTURES: CAN THEY BE OBSERVED ?

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Strong resonant enhancement of magnetic X-ray scattering near the M absorption edges of rare earth elements (0,8 - 1,5 nm) predicted in [1] is shown to result in drastic decreasing of the extinction length for magnetic X-ray diffraction in single magnetic crystals. The extinction length become comparable with the absorption one, and under such conditions it may be possible to detect direct dynamical soft X-ray diffraction on spiral magnetic structures (SMS) (e.g. in metallic Ho, Tb, Dy) in contrast with "satellite" kinematical hard X-ray diffraction on SMS [2].

As follows from our accurate dynamical consideration the X-ray magnetooptics of spiral magnets are similar to the visible optics of spiral liquid crystals (e.g. cholesterics), but the magnetic nature of SMS (gyrotropy) result in more complex dynamical diffraction phenomena. Firstly, X-ray diffraction on SMS shows both "cholesteric" and "gyrotropic" features: two types of reflections may be served at the different Bragg angles relating to "non-magnetic" and "magnetic" periodicity of SMS (half a period and a period respectively). Secondly, these reflections have complex angular and polarization structure: there are a central region of total reflection for any incident X-ray polarization and two regions of selective reflection of a single elliptical polarization; these regions are either in contact or separated by narrow windows of X-ray transparency depending on the values of the gyrotropy and magnetic double-refraction parameters. Thirdly, specific "elliptical" standing waves are formed in spiral magnets over this regions. The polarization control of synchrotron radiation using X-ray diffraction on SMS is realizable. To observe the dynamical diffraction effects the experimental setup must meet the requirements: 1 eV spectral and 10" angular resolutions.

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XRD Characterization of III-V Semiconductor Heterostructures grown on (n11) GaAs Substrates

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In the last years great efforts have been made in the research of GaInAs/GaAs heterostructures due to their importance for optoelectronic application. Most of this work concerned heterostructures grown on [100] orientation. However, new properties are expected when the growth is performed on (n11) substrates. These novel phenomena include larger critical thickness [1,2], corrugated interfaces [3], shear-strained unit cell [4], anisotropy of the band structure [5], and built-in piezoelectric fields [6]. In order to get a deeper insight on these new properties a detailed study is required.

In this paper we present our results on the characterization of InGaAs/GaAs superlattices grown by Molecular Beam Epitaxy on GaAs (n11) substrates. During growth in-situ characterization was performed by Reflection High Energy Electron Diffraction (RHEED). Post-growth characterization by High Resolution X-Ray Diffraction (HRXRD) not only reveal the high structural perfection of our samples, but also confirms the existence of shear strain in the (311)-samples. We compare these results with theoretical predictions [7]. The substrate symmetry strongly influences the relaxation behaviour. (311) superlattices with a large number of periods exhibit strongly anisotropic structural properties and splitting of the satellite peaks not observed in the (100) reference sample.

The electronic structure of these low symmetry semiconductors differs from the (100) counterpart. We present data on the excitonic binding energies in (100) and non-(100) samples.

Our results underline that diffraction techniques reveal several novel phenomena caused by the growth on substrates with low symmetry.

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X-Ray Study of heavily Carbon doped $Ga_xIn_{1-x}As$ ($x \approx 0.99$)

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X-ray scattering methods are of increasing importance for the investigation of epitaxially grown materials. We present an example of how High Resolution X-Ray Diffraction and Topography are applied to study the high carbon doping of GaInAs.

Although Be and Zn have been widely used as p-type dopants of GaAs, it is well known that the diffusion of Be and Zn represents a serious drawback in the performance of devices that make use of these materials. It has been shown that layers of GaAs:C have better properties in terms of the device characteristics. A very high maximum doping level of over 10^{21} cm^{-3} is also attractive. However, the lattice contraction due to C represents an important limitation because of the relaxation of the layers when a critical thickness is exceeded. In order to compensate for this effect we propose the C doping of $Ga_xIn_{1-x}As$ with a small fraction of In. Layers with appropriate C and In concentrations can be grown pseudomorphically on (100) GaAs substrates without relaxation. We have grown a series of $Ga_xIn_{1-x}As$:C layers by Molecular Beam Epitaxy in order to study the incorporation of C and the effectiveness of stress compensation. As C source a graphite filament was used. The doping level was in the range $1x10^{19}$ - $1x10^{20}$ cm^{-3}. X-Ray Diffraction and Topography are used to evaluate the structural quality of the layers. A comparison with Transmission Electron Microscopy characterization is also presented. We have found that C incorporates in the $Ga_{1-x}In_xAs$ lattice on As sites in the range of C concentration studied. The observed deviation in the C concentration evaluated by SIMS and XRD indicates that a certain fraction of the C incorporates either in interstitial sites or as clusters. The density of extended defects revealed by XRT shows the effectiveness in the intended stress compensation.

X-RAY ROCKING CURVE ANALYSIS OF CRYSTALS WITH BURIED AMORPHOUS LAYERS. CASE OF ION IMPLANTED SILICON

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X ray rocking curve simulation of implanted silicon was recently used to investigate the damage accumulation with increasing ion dose [1,2]. For low or medium ion mass, the maximum lattice strain was observed to increase slowly in the low dose range and to undergo a much faster increase when the dose approaches and reaches the value for the formation of a continuous surface amorphous layer.

In a similar investigation here reported, the trend of the integral of the strain depth profile, as determined by rocking curve simulation, was analysed as a function of the ion dose. It was observed that the minimum dose able to produce a buried amorphous layer corresponds to the onset of the regime of fast damage increase. Starting from this dose, the determination of the peak value of the strain depth profile, and hence of its integral value, is meaningless. In fact, by a simple diffraction model it is shown that layer amorphicity can be described by sufficiently high values of the static Debye-Waller factor and that the rigid outward translation of the surface damaged but still crystalline layer with respect to the substrate, induced by the buried amorphous layer, cannot have a definite value. The rigid translation T, given by the product between the thickness of the amorphous layer and the dummy strain value obtained from rocking curve simulation, can be written as $T = (n + x)\, d$, where n is an integer ($n = 0,1,2,...$), $0 < x < 1$ and d is the spacing of the diffraction planes. This implies that, for a given fraction x of d, there exists a discrete infinite set of T values able to give the same calculated rocking curve. The conditions for which this indetermination cannot be solved are discussed.

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DETERMINATION OF LATTICE DISTORTIONS IN EPITAXIAL LAYER SYSTEMS

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The lattice distortions for two GaAs deposits on Si substrates, 3.5° and 1° misoriented off (001) (rotated about [110]) have been calculated from precisely measured diffraction angles. These angles were determined for symmetrically equivalent reflections of GaAs, each four of the type 117 and 115, with a precision of about (1 to 2) × 10^{-3} degrees.

The reciprocal metric tensors G^*_{ik} of the deformed deposits were calculated by solution of the linear equation system

$$(2\sin\Theta_i/\lambda)^2 = (h_i\,k_i\,l_i)\begin{pmatrix} G^*_{11} & G^*_{12} & G^*_{13} \\ G^*_{21} & G^*_{22} & G^*_{23} \\ G^*_{31} & G^*_{32} & G^*_{33} \end{pmatrix}\begin{pmatrix} h_i \\ k_i \\ l_i \end{pmatrix}$$

(Θ diffraction angle; λ wavelength; i = 1, 2, ..., 8). From the six independent components of these tensors, the following parameters of the direct lattice of the deformed deposits are obtained:

	sample no.1		sample no.2	
a/nm	0.566095	± 0.00002	0.566278	± 0.020003
b/nm	0.566109	± 0.00002	0.566220	± 0.00003
c/nm	0.564640	± 0.00002	0.564413	± 0.00003
α/deg.	90.0024	± 0.002	90.0030	± 0.003
β/deg.	89.9953	± 0.002	89.9998	± 0.003
γ/deg.	89.9942	± 0.002	89.9910	± 0.003

From these results, it is obvious that the deformation of the deposit is not tetragonal, but triclinic or monoclinic, respectively. The reason for that is the existence of an epitaxial misorientation between deposit and substrate lattices (see, e.g., [1-3]) in the order of magnitude of 0.1°.

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NEW POSSIBILITIES OF THE RISE OF INFORMATIVITY, SENSITIVITY AND RAPIDITY IN THE HIGH-RESOLUTION TOPOGRAPHY AND DIFFRACTOMETRY

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New possibilities to increase substantially the informativity and sensitivity of high-resolution diffraction methods have been discussed. They connected with the account and use the new physical effects predicted by the authors in their variant of the statistical dynamical theory for single crystals with defects in defects. Namely, there are

— extinction effects in both coherent and diffuse diffraction intensities, which are caused by the scattering on defects;

— effect of the anomalous transmission for the diffuse scattering by static distortions caused by defects;

— violation of the total integrated reflecting power (TIRP) conservation known in kinematical theory in case of dynamically scattered crystals that causes the uniquely sensitivity of TIRP to characteristics of defects. It has been predicted that such sensitivity was possible due to the increasing relative contribution of the diffuse component into the TIRP by the appropriate variation of crystal thickness, radiation wavelength, magnitude of the diffraction vector, azimuthal rotation angle in both Laue and Bragg geometries of diffraction that is important both in diffractometry and topography.

— effects of the non-additive influence of combined distortions caused simultaneously by microdefects and macrodeformation on the TIRP, namely, the loss of the TIRP sensitivity to the deformation (bending) when the defects are present, the possible sign change of their influence on the TIRP value and the increase of TIRP sensitivity to defects with increasing macrodeformation.

All the effects mentioned above have been demonstrated in the paper. The necessity to account these effects for correct interpretation of experimental data and reliable diagnostics of defects has been shown with respect to the traditional methods as well as to the proposed new TIRP methods (so called integral triple-crystal diffractometry) which allow to measure separately diffuse and coherent components of TIRP.

Also, some other new methods that use the new effects have been demonstrated. The established dependences of the effects mentioned on type and characteristics of defects allow to increase substantially informativity and sensitivity of the defects diagnostics.

DYNAMICAL THEORY OF X-RAY DIFFRACTION BY ELASTICALLY DEFORMED SINGLE CRYSTALS CONTAINING DEFECTS

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The classical dynamical theory developed by Ewald, Bethe and von Laue for perfect crystals [1] is generalized for the case of radiations diffraction in macroscopically deformed single crystals containing randomly distributed bounded defects. When obtaining the basic equations for the Fourier transformed amplitudes of wave fields from a wave equation, the method of fluctuation waves of static displacements of atoms was used [2]. The solutions of the basic equations for strong Bragg and diffusely scattered waves have been found in the two-beam case of diffraction by using a perturbation method [3]. The application of these methods in the case of the macroscopically deformed crystal is possible due to the "correcting" change of space coordinates, which provides the translation invariance 'in average" (i. e., after averaging over a random distribution of defects) for the crystal lattice in the new coordinate system.

The dispersion corrections to the wave vectors of strong Bragg waves have been calculated in the two-beam approximation what allows to take correctly the extinction of these waves because of the dynamical diffuse scattering into account. The general expressions for coherent and diffuse components of the differential reflecting power of the deformed crystal with defects have been obtained with taking the boundary conditions for both Laue and Bragg geometries of diffraction into account. The obtained expressions are the absolute squares of the convolutions of the corresponding scattering amplitudes in the underformed defective crystal and a certain weight function which describes the distribution of contributions from various tie points on the dispersion surface into the scattering amplitude and depends on parameters of the macroscopic deformation. The formulae for the diffraction intensities are valid at an arbitrary form of the macroscopic deformation field satisfying only the restriction that its distortion tensor should be small through the whole crystal.

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SYNCHROTRON TOPOGRAPHIC STUDIES OF SINGLE-CRYSTAL SYNTHETIC DIAMONDS

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Synthetic diamonds grown from seed crystals, under conditions of high pressure and high temperature, sometimes contain opaque particles a few micrometres in diameter (called 'clouds'). These are of interest to industry since single-crystal diamonds, after laser drilling, are used in wire-drawing applications.

Lattice misorientations and strains were measured by double-crystal synchrotron X-ray topography and by rocking-curve analysis, turning each specimen crystal through 1 second of arc intervals. A silicon reference crystal reflected 0.96Å radiation (and its harmonics) on to each diamond specimen. The 333 diamond reflexion was well matched by the 800 silicon reflexion in a convenient non-dispersive Bragg-Bragg geometry. (The Bragg angles were 45° and 44.42° for the silicon and diamond respectively.) The topographs were imaged by almost purely monochromatic radiation, since 666 is a 'forbidden' reflexion for diamond and the wavelengths appropriate for higher harmonics were very weak in the synchrotron spectrum.

Individual topographs revealed inclusions, dislocations, boundaries and stacking faults; and series of topographs taken at successive angular positions enabled growth histories of the diamonds to be mapped from their 'cloudy' centres outwards. Rocking curve widths for entire specimens ranged from 5 to 30 seconds of arc (FWHM) for heavily 'clouded' specimens. Rocking curves recorded from areas selected by a translatable pinhole of 100 μm diameter showed that regions of crystal close to the seed had a rocking curve width of typically 5"; and further out there were regions displaying wider (20"), and occasionally narrower (4") rocking curves.

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HIGH RESOLUTION X-RAY DIFFRACTION AND X-RAY STANDING WAVE INVESTIGATIONS OF INDIUM PHOSPHIDE CRYSTALS IMPLANTED WITH Fe^+ .

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Structural distortions due to various dose implantation of Fe^+ ions in semi-insulating crystals of indium phosphide were investigated by means of high resolution X-ray diffraction and X-ray standing wave (XRSW) methods. In frames of XRSW method energy-selective photoelectron emission from samples was measured. As well known /1/ photoelectron yield has high sensitivity to total displacement of near surface atomic planes. Experimental arrangements included a triple crystal X-ray diffractometer and a gas-flow proportional counter of photoelectrons.

For qualitative analysis the mean escape depths of photoelectrons in InP were found (few hundreds angstroms depending on electron energy) by means of measurements of photoelectron yield in grazing-incidence Laue-geometry/2/. For each rocking curve multiple minima corresponding to different distortion profiles were found and some of them provided practically equal values of χ^2 . Each profile was tested by calculating photoelectron yield angular curve and comparing it with the experimental one.

It is shown that XRSW method provides valuable complementary information on strain and damage in the subsurface layer of a crystal and allows to distinguish between different distortion profiles that give practically one and the same kinematical diffraction intensity.

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X-RAY SCATTERING FROM THIN LIQUID WETTING FILMS

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Apparently the structure of the liquid-vapour interface is thickness dependent for a ultra-thin film completely wetting a microscopically rough solid surface. This was studied by x-ray diffraction in the region of total external reflection.

A silicon wafer covered with it's native oxide layer is completely wetted by carbon tetrachloride [1]. Differential heating the substrate surface rerlative to the temperature of a liquid reservoir was used to thin the liquid wetting layer [2].

Both x-ray specular reflectivity and surface diffuse scattering were measured. The data are fitted by a model within the distorted wave Born approximation (DWBA) recently formulated by Holý and Baumbach [3] using **one consistent** set of parameters. The thinnest films are constrained to follow the height variations of the substrate which is modelled by a correlation length perpendicular to the surface. For thicker films this effect becomes weaker [2].

The x-ray measurements were carried out at HASYLAB on the BW1 beamline with a wavelength of $\lambda = 1.3696$Å.

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X-RAY TOPOGAPHIC STUDY OF POTASSIUM NITRATE SINGLE CRYSTALS

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Potassium nitrate, KNO_3, crystallizes in the trigonal-rhombohedral calcite and the orthorhombic aragonite structure. The aragonite structure is stable below 128°C with lattice parameters **a** = 5.42 Å, **b** = 9.17 Å, **c** = 6.48 Å and space group Pmcn (historical non-standard setting). Large single crystals (up to 50 mm diameter) of aragonite-type KNO_3 were grown from aqueous solution at about 40°C by slow evaporation of water. The crystals tend to develop parallel intergrowths with re-entrant edges and the capture of liquid inclusions. Occasionally thin pseudohexagonal {110} twin lamellae, typical for aragonite, are observed with polarized light and by etching.

Slices (about 1 mm thick) parallel (100), (010), (001) and (110) were cut out of the crystal with a string saw and water as solvent and polished on a soft cotton cloth soaked with water. The slices were studied by conventional X-Ray topography (LANG technique) using MoKα radiation of a rotating-anode generator.

The topographs show that a rather wide zone surrounding the seed crystal is strongly disturbed. Some peripheral regions, however, are highly perfect, containing only a few dislocations. Straight-lined grown-in dislocations with directions close to the normal of the growth face concerned or strictly parallel to directions [001] and [110] originate from the disturbed regions. Some of them show post-growth movement by glide. Burgers vectors [100], [010] and [001] have been identified. In addition, numerous glide dislocations with Burgers vector [100] (shortest lattice translation) are present, indicating the highly anisotropic plasticity of KNO_3. Most of them have the shape of halfs-loops parallel to the pseudohexagonal (001) plane. Frequently they are arranged in columns of half-loops parallel [100], the direction of their Burgers vector. The glide is blocked by the twin lamellae. Thus glide dislocations accumulate along twin lamellae which fequently separate regions of high glide-dislocation density from dislocation-free regions. Grown-in dislocations are observed to penetrate the twin lamellae.

The support of this study by the **Stiftung Volkswagenwerk** is gratefully acknowledged.

HIGH-RESOLUTION MAPPING OF TWO-DIMENSIONAL LATTICE DISTORTIONS FROM TRIPLE-CRYSTAL X-RAY DIFFRACTOMETRY DATA

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Lattice distortions perpendicular to the surface in thin surface layers of ion-implanted (111) silicon crystals have been mapped as a function of depth and lateral position with submicron resolution. The samples are implanted with 300 keV B^+ ions to a 5×10^{15} cm^{-2} dose through a one-dimensional strip mask oxide pattern with a 5.83 μm repeat period and a 4 μm gap region, which produced a series of satellite peaks in rocking curves observed near the 111 Bragg reflection. Small areas in reciprocal space were scanned using a triple-crystal X-ray diffractometer at a synchrotron source. The intensity data were treated by an algorithm [1] which Fourier synthesized *ab-initio* solutions of the 1D inverse problem [2]. Data collected on the vertical-wiggler beamline at the Photon Factory to achieve 0.33 μm in-plane and 500 Å depth resolution in the resulting maps show a very thin damaged layer of 0.15 μm thickness at 1.05 μm below the surface, in agreement with Monte Carlo calculations, with increased lattice spacings normal to the surface by several parts in 10^4. The distortions are appreciably extended in the lateral direction suggesting diffusion of the enegetic ions. The 0.5 μm-thick thermal oxide strip is found to contract the normal lattice spacing of substrate silicon crystal under the strip region by a few parts in 10^4 because of the built-in surface tension [3]. The model distortions were checked by back computing rocking curve profiles using the Takagi-Taupin formula. The use of a four-bounce channel cut for the analyzer crystal on the BIGDIFF diffractometer at the Australian National Beamline Facility led to a reduced background, allowing one to scan far from the Bragg peaks for significantly improved in-plane and depth resolutions of 0.22 μm and 160 Å respectively in a surface layer of 1 μm thickness. A further improvement in signal-to-background ratio was achieved on evacuating the diffractometer down to 1 Torr, which promises a depth resolution of 50 Å in the maps [4].

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X-ray topography study of Highly Oriented Pyrolytic Graphite

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Highly Oriented Pyrolytic Graphite (HOPG) is a very efficient and well-known X-ray and neutron monochromator [1], obtained by thermal cracking of a hydrocarbon gas and subsequent graphitization treatment [2]. This process leads to a rotational disorder of the graphite layers, which have only in common the c axis of the hexagonal structure. But the actual microstructure is still a matter of controversy. Length and depth along the c axis of the 'crystallites' extend, when consulting literature, over a wide range (from about 0.01µm to 100µm). From a diffraction properties point of view, the model consistent with most of the published results is that of the „ideally imperfect mosaic crystal" [3], very far from the nearly perfect crystals usually investigated by topographic techniques.

Several samples of different qualities (mosaic spread of the c axis going from 18' to more than 1°) and provided by different manufacturers were characterized by a set of experimental techniques. These included diffraction (Laue technique, high resolution diffractometry) and topograhic techniques (Lang's method, synchrotron radiation topography - at LURE - performed either by recording 'projection' topographs, or by using the 'section' and even 'pinhole' versions of this technique, which appear to be useful in our case). Most of the investigations were carried out using the only 'single crystal' reflections produced by HOPG samples, the (000l).

The HOPG topographs show that: 1) bright 'spots' are present within the sample image, elongated along the diffraction vector, 2) these 'spots' do not vary when the topographs are recorded on several points of the (wide, about 0.5°) rocking curve, 3) the obtained images are very dependent on the sample to film distance, the 'spots' divergence being bigger than the average mosaic spread of the sample.

The set of obtained experimental facts cannot be explained by models based exclusively on misorientation distributions of the reflecting planes. An interpretation in terms of 'extinction' appears sensibe: relatively well crystallized growth tubes are seperated by more defective borders of a higher reflectivity. This model can easily be related to the microstructure of as-deposited Pyrolytic Carbon, the departure material for the production of HOPG. In the light of these results, additional information can be gained on the interlayer spacing of the graphite netplanes that is found to remain constant over distances of several micrometers. This may be discussed in the context of graphitization mechanisms.

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Reciprocal space mapping utilising a linear position sensitive detector

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With regard to structural information of epitaxial layers (lattice strain, relaxation, mosaicity, layer thickness...) so-called reciprocal space maps provide the most information. Reciprocal space mapping is usually carried out by a triple axis arrangement in which the direction of the diffracted beam is measured either with (a) a crystal analyser or, alternatively, with (b) a small slit located in front of the detector. The mapping is performed by a combined scanning of the sample and the detector in such a way that the scattering vector **Q** covers an area in reciprocal space ("area scan"). Both methods, however, differ substantially concerning resolution and intensity. Depending on the slit width, method (b) provides a resolution of about $\Delta Q/Q \approx 10^{-4}$ ... 10^{-3} which is limited by the beam size. An analyser, on the other hand, improves the resolution to 10^{-5}... 10^{-4}. However, the high resolution achieved is accompanied by a strong intensity reduction due to the small angular acceptance of the crystal analyser.

The investigation of very thin epitaxial films (with thicknesses of a few atomic layers) by conventional reciprocal space mapping as described above is rather time consuming, in particular, when asymmetrical reflections are investigated. We present here a novel experimental method using a linear position sensitive detector (PSD) instead of a crystal analyser (see figure below). Diffracted beams within 0.5° .. 1° divergence angle can be measured simultaneously. Due to the spatial resolution of the detector (100 µm) a moderate resolution down to 10^{-4} can be achieved. In order to take advantage of this resolution the horizontal size of the diffracted beam should be around 100 µm. For this reason an asymmetrical geometry with glancing exit is most advantageous. Multi-detection and moderate resolution combined with a 12-kW rotating anode generator (line focus) ensures a considerable enhancement of intensity as compared with conventional reciprocal space mapping.

The experimental setup is most suited for systems with large lattice mismatch between layer and substrate, thus requiring a large angular range and not too high resolution. Preliminary results of 5 and 10 nm Ge_xSi_{1-x} LPE layers grown on Si 001 (with x around 0.85) will be discussed and compared with results obtained with a triple crystal arrangement.

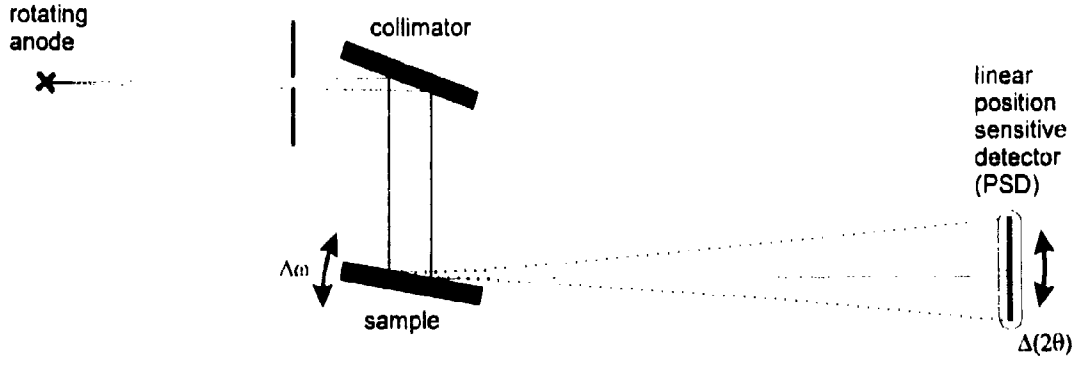

Fig.1: *Schematic view of the experimental setup in a (m,-n) scattering geometry. The sample and the detector are mounted on a two-circle goniometer.*

X-RAY DIFFRACTION DIAGNOSTICS OF LASER HETEROSTRUCTURES

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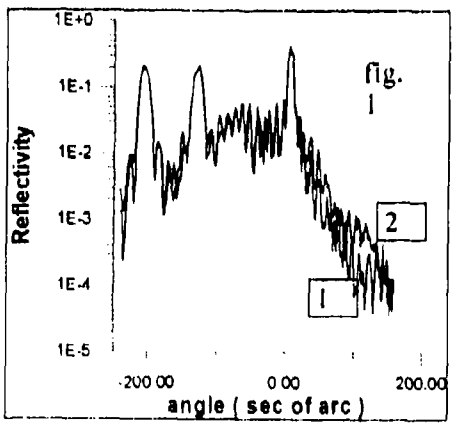
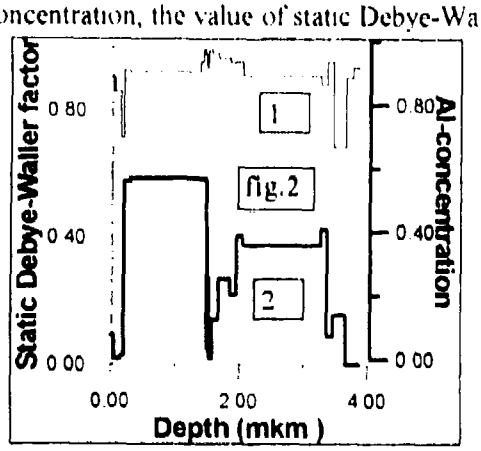
Usually X-ray diffraction diagnostic of multilayer systems is carried out without taking into account incoherent scattering caused by defects. The separation of coherently and incoherently scattered intensity enables to obtain the information both on the depth-dependent strains and statistically distributed microdefects. The inverse problem of X-ray diagnostics of heteroepitaxial laser structures is solved in the framework of the statistical dynamical diffraction theory, coherent and incoherent parts of scattered intensity being taken into account. The N-layer system model represents alternating crystalline layers. Each of the layers, say with number n, is characterized by an intrinsic interplanar spacing d_n , the static Debye-Waller factor E_n and average size of defects r_n. In solving the inverse problem the laser structure parameters (AlGaAs) were found by means of misfit functional minimization



$$R = \sum_{i=1}^{k}\left(R_i^e - R_i^t\right)^2 + \sum_{i=1}^{k}\left(R_{i,c}^e - R_{i,c}^t\right)^2 = R(\bar{X},\bar{E},\bar{r})$$

where $R_i^{e,t}$ are experimental and theoretical double-crystal scheme reflection coefficients, $R_{i,c}^{e,t}$ are reflection coefficients a triple-crystal $\theta-2\theta$ scanning scheme; k is the number of experimental points, $\bar{X},\bar{E},\bar{r}$ - are (n+1)-dimensional vectors determining Al concentration, the value of static Debye-Waller factor and the mean defect radius in each layer. Functional minimization of the above type involves considerable computational difficulties due to the multiplicity of local minima, a large number of parameters and the functional nonlinearity. A direct search method was employed, restrictions being imposed on vectors $\bar{X},\bar{E},\bar{r}$ defined. The profiles of the depth-distribution Al-concentration , the static Debye-Waller factor and the size of defects were obtained. Fig.1 displays calculated (1) and experimental (2) triple-crystal reflection curves. The profiles of static Debye-Waller factor (1) and Al-concentration (2) are shown in Fig.2.



NUMERICAL IMAGE TREATMENT OF SYNCHROTRON WHITE BEAM TOPOGRAPHS

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The analysis of synchrotron white beam topographs can be greatly improved using numerical image treatments.
On line digitization and simple treatments during the experiment allow to greatly improve the quality of the TV images and to follow images changes during the experiment. For instance it has been possible to study the interaction of dislocations with surface acoustic waves.
Fourier filtering and wavelet analysis are efficient tools to restore and enhance the quality of topographs. They allow to extract more information than provided with just a visual analysis of the original topograph.
We have used these treatments to process topographs of surface acoustic waves devices and ferromagnetic domains in Fe-Si crystal.
Background correction and feature enhancement with polar filters make possible the study of the distortion of the shape of the acoustic wave due to crystal defects. The losses of energy in surface acoustic device are also clearly visible in the processed topographs.
In Fe-Si crystal topographs the contrast due to ferromagnetic domains are masked by the disorientation contrast of subgrains. Low-cut frequential filters and anisotropic filters greatly enhance their visibility, especially at the subgrain boundaries.
A program is now available for images treatment and its main features will be explained in a further paper.

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DYNAMICAL X-RAY DIFFRACTION FROM MULTILAYER SYSTEMS WITH DEPTH-DEPENDENT DISTRIBUTED MICRODEFECTS

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Dynamical of X-ray diffraction from a nonuniform crystal with depth-dependent strain, concentration and size of defects is formulated by recursion formulae. The amplitudes of the transmitted E_0^c and the diffracted E_g^c coherent waves for layer with number n ($1 \le n \le N$, N is number of layers in the multilayer system) in the Bragg case are

$$E_0^{(n)}(z) = \left[T_n/(S_1 - S_2)\right]\cdot\left\{S_1 \exp\left[i\varepsilon_2(z - z_n)\right] - S_2 \exp\left[i\varepsilon_1(z - z_n)\right]\right\}$$

$$E_g^{(n)}(z) = \left[T_n/(S_1 - S_2)\right]\cdot\left\{S_1 b_2 \exp\left[i\varepsilon_2(z - z_n)\right] - S_2 b_1 \exp\left[i\varepsilon_1(z - z_n)\right]\right\}$$

where z_n is the coordinate of the layer with number n, $S_1 = (R_{n-1} - b_1)\exp(i\xi l_n)$, $S_2 = (R_{n-1} - b_2)$, l_n is the thickness of this layer, $b_{1,2} = \xi_{1,2}/E_n\sigma_g$, where E_n is the static Debye-Waller factor, $R_{n-1} = E_g^{(n-1)}(z_{n-1})/E_0^{(n-1)}(z_{n-1})$ is the amplitude reflection coefficient for the lower $(n-1)$ layers in total; $\varepsilon_{1,2}$ are the angular variable (other notations see in [1]). T_n is the amplitude transmission coefficient for the upper $(N-n)$ layers in total. For N-layer system the angular distribution of the incoherently scattered intensity emerging from the layer with number n is given by the recursion formula

$$I_n^i = I_{n-1}^i \exp(-\mu l_n) + \left(\frac{2\left|\sigma_g\right|^2\left(1 - E_n^2\right)\tau_n\left|T_n\right|^2}{\left|S_1 - S_2\right|^2}\right)\left\{\left|S_2\right|^2 a_1 + \left|S_1\right|^2 a_2 + 2\,\mathrm{Re}\left(S_1 S_2^* a_3\right)\right\}$$

where I_{n-1}^i is the incoherently scattered intensity from the lower $(n-1)$ layers in total (notations for a_i $(i = 1,2,3)$, τ_n and σ_g see in [1]). Numerical calculations for theoretical rocking curves and the distributions of depth-dependent coherently and incoherently scattered intensities are made for the nonuniform system $Al_xGa_{1-x}As/(100)GaAs$, where depth-dependent concentration x is $x(z) = \left[x_0 - \left(\frac{\Delta x}{l}\right)z\right]/(1 + z/l)$, l is the thickness and z is coordinate directed into the depth of this system.

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THE SPACE AND ANGULAR DISTRIBUTION OF X-RAY INTENSITY IN LAUE DIFFRACTION IN REAL CRYSTALS

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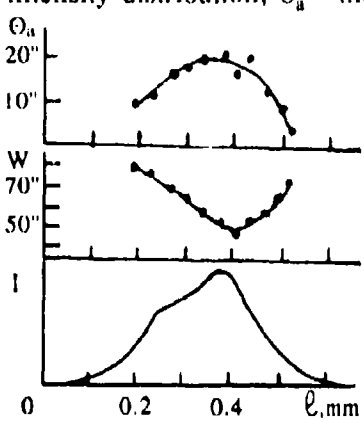
It is recognized that in the limits of the kinematic scattering model of the crystal the refractive index of X-rays is constant and equals 1. According to the dynamical theory the refractive index of X-rays varies in X-ray diffraction in perfect crystals in the Bragg scattering curve region which allows one to determine the dependence between the incident and scattering angles. The behavior of refractive index of X-rays in diffraction in real crystals is of significance. The objective of the present work is experimental study of space and angular distribution of X-ray intensity in Laue diffraction in real crystals as a function of angular deviation (ϵ) from the exact Bragg position. The investigation was carried c . on lithium fluorine single crystals with various dislocation density (N_d). The study was made on double and triple crystal spectrometers in the Cu $K_{\alpha 1}$ radiation, with perfect germanium crystals (reflection 333) serving as the monochromator and analyzer. The experiment consisted in the following. The slit moved sequentially in the horizontal direction across the diffraction beam and was fixed at certain point *l*. In this case a region was picked up from the diffracted beam cross-section at the given ϵ value for which the direction of diffraction (Θ_a) was determined with the help analyzer. These experiments allowed us to determine also the space distribution of the diffracted beam intensity. Fig. shows, by way of example, the results of investigation for $\epsilon = 0$ and $N_d = 3 \cdot 10^6$ cm^{-2}, where I - the diffracted beam intensity distribution, Θ_a - the angular position of the center of gravity of reflection curve of the analyzer, W - the integrated width the reflection curve of analyzer. The analysis of the results obtained showed that the integrated characteristics change essentially depending on ϵ and *l*. Thus, at the given value of the deviation angle from the Bragg position for various regions of diffracted beam the X-rays are scattered at different angles. This evidences that for different ranges of real crystal at the given value of ϵ the X-rays have different refractive indices. The change of refractive index has been estimated for different ϵ values.



DEFECT STRUCTURE OF CAST SILICON AND OVERGROWN HOMOEPITAXIAL LPE LAYERS

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Cast Silicon is among the many substances which are tested in search for low cost solar energy conversion. We have accompanied attempts to grow thick silicon LPE layers with improved electrical parameters on two types of cast material: SILSO (10^{16} B · cm^{-3}) and SITIX (10^{19} B · cm^{-3}).

Both materials consist of randomly oriented grains ranging from a few tenths to several 10 mm^2 in lateral dimensions. Most grains contain twins in varying numbers and sizes. The average dislocation density is $10^5 - 10^6$ · cm^{-2} within the grains. In SIlSO the dislocations are often arranged in a cellular structure and in small angle boundaries as revealed by XRT and by etching. In SITIX elongated dislocations arranged in bushels and bundels are prevailing which, according to TEM, are helical in nature and decorated with particles.

The dislocation arrangement extends practically unchanged into the Epilayer for SILSO. No helical dislocations are observed in layers grown on SITIX. Due to the high dopant concentration of this latter substrate a high density of misfit dislocations is generated occasionally.

In layers grown with low supersaturation single dislocations do not generate EBIC contrast.

X-RAY DIFFRACTION DISTINCTIONS IN THE ACOUSTICALLY EXCITED CRYSTAL WITH MICRODEFECTS.

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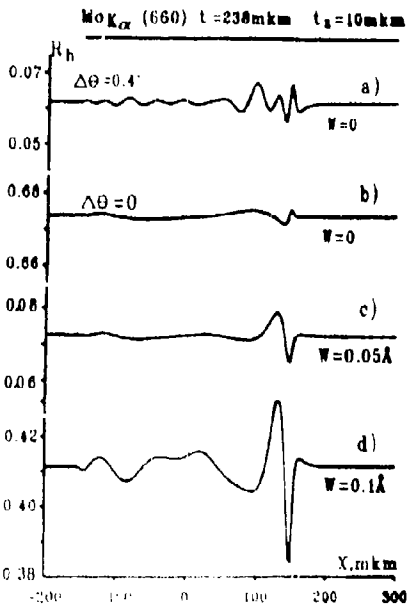
On the base of results, determined from Takagi equations numerical solutions,is shown the opportunity of ultrasound utilization like off-beat sounder for contrast increasing of microdefects, not visible in common conditions ($r_0 = 0.16$ mkm), and its diffraction region size increasing. It is shown, that the ultrasound excitation can substantionally change the diffraction image size (Fig.1). This can be attributed by the intensity redistribution inside the crystal in dependence of amplitude value W, that couses the microdefects diffraction image change. When W= 0.05 Å one can see the diffracted beam background intensity decreasing by 10 times and t' e microdefect direct image contrast V ($V=(R_{max}-R_{min})/(R_{max}+R_{min})$) increasing from 0.3% to 10% (Fig. 1b,c). When W=0.1Å one can see R_h decreasing by 15% and V increasing up to 1.5% (Fig.1d). On t. : Fig.1c diffracted wave intensity reachs the same limits, that during excitation point evaluation on the hill of the reflecting curve (Fig.1a), where V has the same value (~9%). Besides, at given W value the image direct component contrast reversal occurs. Consequently the X-Ray diffraction intensity oscillating dependence on ultrasound amplitude can be utilized for microdefects image fine structure reveal and contrast increasing.



X-RAY TOPOGRAPHIC INVESTIGATION OF α-$LiIO_3$ UNDER DC OR AC ELECTRIC FIELD

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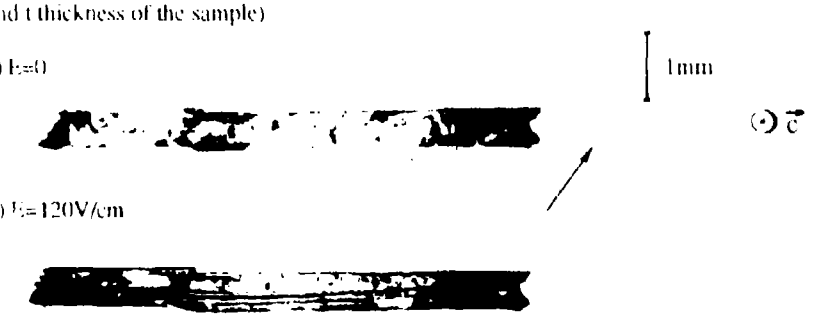
Several single crystals such as quartz, KDP,... change their behaviour if a static electric field is applied. This is also true, even in a more spectacular way, for single crystals of α-$LiIO_3$. Under an electric field (10^2-10^3 V/cm) applied parallel to the c-axis, the Bragg diffracted intensity for neutrons is multiplied by a factor varying from 2 to 10 and enhancements in optical diffraction and changes in dielectric behaviour have been reported [1,2].

The X-ray diffracted intensity of some Bragg reflections is also modified by the application of an electric field (an enhancement of the diffracted intensity in the low absorption case) and traverse topographs show many fine lines parallel to the c-axis. Previous investigations [3,4] of this behaviour under different experimental conditions suggest a mechanism for this electric field induced reduction of crystalline perfection. The ionic conductivity of α-$LiIO_3$ is non homogeneous over the crystal and occurs through "channels" parallel to c-axis. A gradient of lattice distortion is established between these channels and the remaining part of the crystal, leading to a modified Bragg reflectivity.

Up to now the enhanced reflectivity was related to the fine lines on topographs. The present work based on section topographs (fig.1) shows, that these striations are only a secondary effect. The majority of the enhanced diffracted intensity appears to come from small areas of crystal which form 'planes' nearly parallel to the c-axis and the main surfaces of the sample and which seem to correspond to growth bands.

The influence of the electrodes used is also discussed. We finally describe and compare the results of topographic experiments and conductivity measurements under an alternating electric field, which are in qualitative agreement.

Fig.1: Section topographs of 137 reflection, λ=0.4Å, μt=2.2 (μ is absorption coefficient and t thickness of the sample)



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SI-TASI$_2$ COMPOSITE STUDIES BY SYNCHROTRON RADIATION WHITE BEAM TOPOGRAPHY AND MULTIPLE-AXIS DIFFRACTOMETRY

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The eutectic composite $TaSi_2$/Si can be grown in the form of a single crystal Si matrix with rods of the silicide phase grown parallel to the crystal growth direction. The rods generally have a high degree of preferred orientation relative to the matrix, and the matrix is highly dislocated (by bulk silicon standards). Rod diameters of about 1μm and inter-rod spacings on the order of 10μm are typical in the $TaSi_2$/Si system, and double crystal rocking curves of the Si matrix are many times broader then those of dislocation-free Si. On a scale of 100μm and above, however, the composites are suprisingly uniform. This suggests that $TaSi_2$/Si crystals might be useful as wide-band pass x-ray monochromators or analysers.

In this paper we report characterization studies of a number of $TaSi_2$/Si composites and also preliminary results obtained using the composites as monochromators or analyzers. The composites have been examined to date with techniques which include polychromatic and monochromatic x-ray diffraction topography, x-ray double axis diffractometry and triple axis diffractometry.

A VIBRATING MONOCHROMATOR FOR THE E.S.R.F. 'TOPOGRAPHY' BEAMLINE

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The small size of the sources (≈0.2 mm), the small divergence (≈10^{-4} in the vertical plane) and high flux of the X-ray beam are prominent features of the ESRF. This opens new possibilities for diffraction topographic techniques. The way we have chosen to take advantage of these features is to build a long (140 m), wiggler, beamline, the ID19 E.S.R.F. "Topography" station [1], allowing a homogeneous beam of ≈40 mm * 14 mm.

It appears under these conditions that the exposure time to record a topograph (≈0.1 sec) will be considerably reduced when working at the ID19 station. In addition both the angular divergence $\delta\theta$ and spectral spread $\Delta E/E$ of the monochromatic beam reaching a point of the sample will be < 10^{-5}, even when using a simple monochromator (as opposed to a multiple crystal one). These $\delta\theta$ and $\Delta E/E$ are very convenient for some applications, but not big enough for others (slightly distorted crystals, phase transitions,...). In addition the diffracted energy is a function of the position within the monochromatic beam section ('wavelength dispersion').

A solution to overcome the insufficient range of energy and the wavelength dispersion, is to change periodically, as a function of time, the Bragg angle θ_B of the monochromator. The oscillation of the monochromator around the vertical axis should be such that several periods are included in the exposure time: this implies a frequency range up to 200 Hertz, a value which is substantially higher than previously [2].

We have tested several options to vibrate the 0.3 mm thick silicon monochromator diffracting in the transmission 'Laue' mode. A piezoelectric device appears to be unable to give the required amplitude, and heats when used during extended periods (≈ 10 minutes). A 'door bell' like coil solves the amplitude problem but presents a non reproducible rest position, partly due to the friction. We retained a vibrating device based on a triple pole electromagnet and a $SmCo_5$ permanent magnet (fig.1). The initial monochromatic beam topographs show that this device exhibits the expected performances both for the amplitude and the reproductibility of the 'zero' rest position.

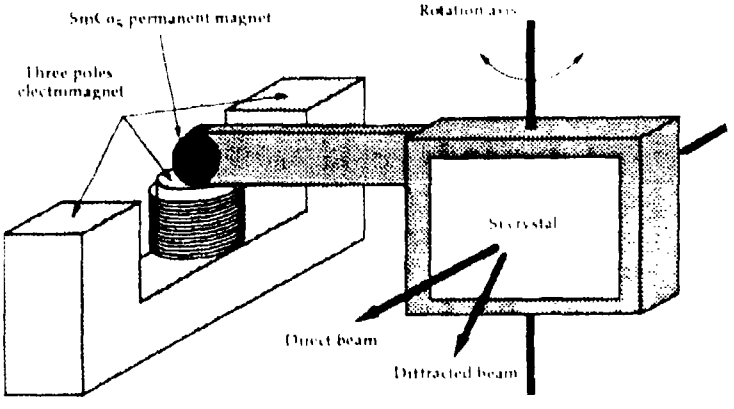

Fig. 1

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INVESTIGATION OF THE RELAXATION BEHAVIOUR IN STRAINED SUPERLATTICES USING GRAZING INCIDENCE DIFFRACTION OF SYNCHROTRON RADIATION

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Lateral lattice matching is only possible up to a critical thickness t_{cr}, beyond which misfit dislocations are created at the interfaces. For single layers of $Ga_{0.8}In_{0.2}As$ on GaAs it amounts to 19 nm. In the case of superlattices (SL) the critical thickness depends on 1) the In- content, 2) the total thickness of sl $t_{tot} = (t_a + t_b) n = t_{SL} n$ (t_{SL}: SL- period, n: number of periods) and 3) the thickness ratio t_a / t_b.
In order to unterstand the mechanism of relaxation we have investigated the dependence of t_{cr} on the thickness of the barriers t_b. The real structure of the SL was investigated measuring conventional rocking curves and reciprocal space maps. The in-plane parameter were measured using grazing incidence diffraction of synchrotron radiation. The depth dependence of the relaxation degree can be determined by measuring the rod scans of the in-plane Bragg-peaks at different angles of incidence [1]. The shape of the rod-scans is influenced by the real structure. The amount of the real-structure-effects is correleted to the degree of relaxation. Because of the imperfections a kinematic description of the experiments is sufficient.
Although the thickness of the $Ga_{0.8}In_{0.2}As$ layers were chosen smaller than the critical one, partial relaxation of the superlattices was obtained for barrier thicknesses between 2 nm and 30 nm. In contrast to our former investigations [2] we found a homogenous relaxation state over the whole depth of superrlattice.

This work was supported by the BMFT No 05 5IPAA1 8.

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DYNAMICS OF ROCKING CURVES IN STRAINED (001) Si CRYSTALS UNDERGOING ULTRASONIC EXCITATION

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Dynamical X-ray diffraction under high-frequency ultrasound (US) was recently found to be very sensitive to small strains in almost perfect crystals [1]. US with a wave vector |k| larger than the characteristic gap of the dispersion surface stimulates interbranch scattering processes that lead to various modifications in the diffraction profiles, depending on the strain level ε of the sample and on the US amplitude w. When the dynamic strain $\delta=2|k|w$ induced by US exceeds the intrinsic strain ε, a broadening of the diffraction profile is expected due to satellites formation on the acoustic superlattice. In the opposite case, $\delta < \varepsilon$, the interbranch scattering processes reject part of X-rays from the diffracted beam, resulting in a narrowing effect of the diffraction profile. This narrowing effect was first observed by us in double-crystal diffraction (DCD) measurements of rocking curves in slightly deformed Si crystals [2]. Here we present results of the rocking curves shape dependence on the US amplitudes.

DCD measurements were carried out in the Bragg geometry with CuK_α-radiation on (001)Si. The samples were attached to a $LiNbO_3$ piezotransducer by hardened photoresist (with overlapping surfaces as shown in the insert in Fig.1). The transducer generated surface acoustic waves with a resonant frequency of 293 MHz. (004)Si rocking curves were taken at different applied voltages (V) on the piezotransducer. The dependence of the rocking curves width Γ on the applied voltage (V) is shown in Fig.1. Γ(0)-value corresponds to a strain level of $\varepsilon \approx 2 \cdot 10^{-5}$ introduced by the gluing procedure. Γ vs. V clearly demonstrates the narrowing-broadening transformation of the rocking curves under US influence. This result is of a great importance for the establishment of a new strain analysis method based on combined use of DCD with US.

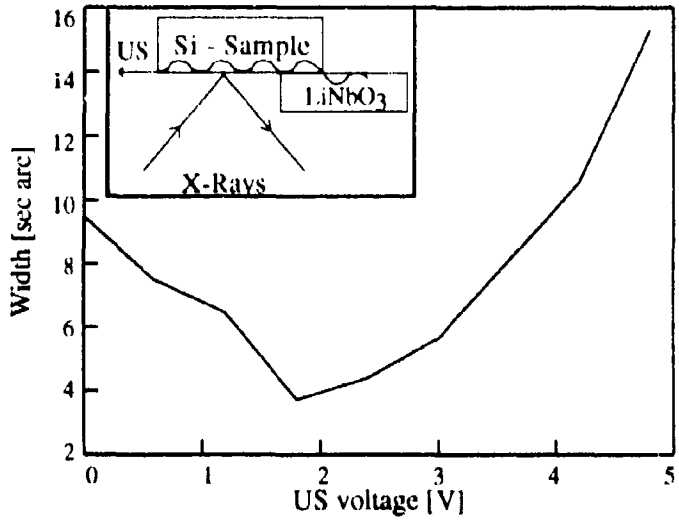

Fig.1. The width of (004) Si rocking curves under US excitation

Insert: sample-transducer set up

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A novel experimental setup for grazing incidence and extremely asymmetrical x-ray diffraction

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Grazing incidence diffraction and specular x-ray reflectometry (including offspecular diffuse scattering) are now two standard techniques which are extensively applied to structural characterization of thin films and interfaces. The combined use of both methods makes it possible to obtain

(i) 'density' information (layer thicknesses, thickness fluctuations, vertical density profiles),

(ii) structural information on mesoscopic scale (interfacial and surface roughness, correlation lengths) and, finally,

(iii) structural information on atomic scale (lattice parameters and relaxation; depth sensitive measurements).

We present here an experimental setup for grazing incidence diffraction, extremely asymmetrical x-ray diffraction and specular/offspecular x-ray reflectometry. However, three main differences as compared to previous scattering geometries are present

- we use the entire *horizontal line focus* of a rotating anode (12 kW, the dimensions of the focus are typically 0.5 mm × 5 mm) without any further geometrical reduction by slits. This geometry applied on a horizontally mounted sample ensures a considerably higher intensity without loosing vertical collimation.
- the collimator system can be turned around the diffracted beam. We are, therefore, able to collimate the incidence beam with respect to various spatial directions. The collimator system consists either of a symmetrical Ge channel-cut combined with a soller slit or of an asymmetric Bartels collimator (6" angle of incidence) with two miscut Ge 220 channel-cuts. Due to simulations both geometries should yield to comparable count rates and resolution.
- extremely asymmetrical reflection geometries (grazing incidence and oblique exit) are also accessible. Thus, it is possible to investigate relaxation in different spatial directions.

The design and the capabilities of the instrument will be discussed with regard to applications.

THREE CRYSTAL DIFFRACTION STUDIES ON OXYGEN INDUCED DEFECTS IN ANNEALED CZ SILICON CRYSTALS

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In Czochralski (CZ) grown silicon oxygen is the most important impurity. Upon heat treatment at 750°C oxygen atoms diffuse through the lattice to produce small agglomerates which grow into precipitate particles of silica (SiO_2). Further annealing at 1050°C or 1200°C leads to the generation of dislocation loops and/or the formation of stacking faults. Three series of CZ silicon crystals containing $7.5, 9.9, 18.1 \cdot 10^{17} cm^{-3}$ oxygen atoms were measured. Three samples of each series had been annealed for 24h, 72h, 216h at 750°C, two samples had been preannealed for 24h at 750°C and then further annealed for 20h at 1050°C or 1200°C.

The size and number density of the SiO_2 precipitates have been determined with Small Angle Scattering (SANS) at the ILL, Grenoble [1]. In addition to SANS the defect scattering was mapped out in **k**-space at the same samples by means of the high resolution three crystal diffractometer for high energy synchrotron radiation installed at the HASYLAB wiggler laboratory. The diffuse scattering was measured with 100 keV synchrotron radiation at reflection 220 in its projection onto the [110]-zone.

The diffuse scattering measured at the samples annealed for different periods of time at 750°C was identified to be related to SiO_2 precipitates [2]. Nearly all samples showed additional diffuse streaks of scattering along the [001] direction. The FWHM of those streaks is increasing with the distance to the reciprocal lattice point. According to Larson&Schmatz [3] dislocation loops on {111} planes with Burgers vectors in ⟨110⟩ directions could cause this diffuse scattering. Their diameters are of the order of 1000 Å.

Diffuse streaks along ⟨111⟩ directions were found in the samples preannealed at 750°C and then further annealed at 1050°C or 1200°C. Along the [111] direction the FWHM of the streaks is constant. The asymmetry of the diffuse scattering in the [220]-zone indicates, that these defects are of extrinsic nature. Model calculations [3] identified these streaks as diffuse scattering from stacking faults lying on ⟨111⟩ planes. Their diameters depend on the annealing history of the sample and vary from about 10000 Å up to 40000 Å.

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High-Resolution X-Ray Diffraction and X-Ray Standing Waves Analyses on AlAs/GaAs Short-Period Superlattices

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The state-of-the-art approach to study the structural parameters of superlattices (SL) is (high-resolution) X-ray diffraction ((HR)XRD) which measures the elastically scattered photons. This method can be extended by measuring simultaneously inelastic signals excited internally by the X-ray standing wave (XSW) field which is generated in the reflection process. Recording element specific inelastic signals provides a unique way to determine the position of the excited atoms. We have applied the measurement of the element inspecific but more intense photocurrent for the structural characterization of the superlattice.

The measurements were performed with 5.30 keV and 8.05 keV photons from the synchrotron radiation source SPEAR at SSRL using a double crystal monochromator. The well collimated and monochromatized synchrotron radiation beam of high intensity enabled us to extend the HRXRD measurement to a wide angular range showing the complete set of satellites between neighboring substrate reflections. Thereby, a non-coincidence of the AlAs/GaAs satellite groups belonging to GaAs(002) and GaAs(004) is observed, which results from an incommensurate modulation of the superlattice. By comparison with kinematic diffraction theory, from the satellite intensities, the Fourier coefficients of the modulation can be determined. This gives a straightforward description of the AlAs/GaAs superlattice unit cell and its interfaces.

The simultaneously measured photoelectron yield shows a clear modulation due to the GaAs(004) substrate reflection, the 0th order AlAs/GaAs SL reflection and the thickness fringes. At the 0th order SL reflection the wave field is aligned to the average lattice plane distance of the AlAs/GaAs SL. The modulation of the photocurrent is a measure for the ratio of coherently and incoherently positioned atoms with respect to the average lattice plane distances. The shape of the photocurrent signal at the GaAs(004) substrate reflection is caused by a moiré effect which leads to a different shape compared to the shape of the 0th order SL reflection. The moiré effect turns out to be present in many heterostructures. It offers a further criterion to differentiate between layer and substrate peaks. The XSW measurements are compared with dynamical calculations of the depth dependent X-ray wave field.

Two of us (G.M. and M.S.) are grateful to the Volkswagen Stiftung for sponsoring theses studies through a grant to Stanford University.

GROWTH AND REAL STRUCTURE OF UREA CRYSTALS

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Urea is a promising material for non-linear optics. Crystals have been grown from methanol solution by reduction of temperature in the range 45-25°C.The average growth rate along the Z axis was within 0.15-0.25 mm/day.

Real structure of crystals was studied by X-ray projection topography (Lang method). Zonal and sectorial structure as well as surface morphology and arrangement of dislocations in urea correspond to growth by dislocation mechanism. Almost all grown-in dislocations observed were of mixed type (see table). There are dislocation-free areas in the central upper part of the crystal. Stresses arising during growth raise redistribution and interactions of dislocations.

The step-like change of the growth temperature of 0.05°C followed by formation of macrosteps and liquid inclusions. They can act as local sources of secondary defects - slip dislocations generated in {110}, {111} and {001} planes of easy slip. Frank-Read sources have been observed in {111} slip planes.

GROWN-IN AND GLIDE DISLOCATIONS IN UREA CRYSTALS

N	Type of dislocation	Angle to C axis,°	Burgers vector	Growth sector	Remarks
1	mixed	12-15	001	{111}	grown-in
2	mixed	31	?	{111}	grown-in
3	mixed	14	$<111>,<\bar{1}\bar{1}1>$	$(111),(\bar{1}\bar{1}1)$	grown-in
4	mixed	68	?	$(111),(\bar{1}\bar{1}1)$	grown-in
5	edge	90	$<110>,<\bar{1}\bar{1}0>$	$(111),(\bar{1}\bar{1}1)$	grown-in
6	edge	90	$<\bar{1}10>,<1\bar{1}0>$	$(111),(\bar{1}\bar{1}1)$	glide
7	52°		$<110>,<1\bar{1}0>$	$(111),(\bar{1}\bar{1}1)$	glide
8	?	?	?	$(111),(\bar{1}\bar{1}1)$	glide,{110}
9	?	90	?	$(111),(\bar{1}\bar{1}1)$	glide,{001}

It follows from topographs observed that the stability of the temperature during growth should be better than 0.01° C for growing crystals of high quality enough.

X-RAY TOPOGRAPHY OF FACETS IN GaSb SINGLE CRYSTALS

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The <111> GaSb single crystals grown using the Czochralski technique without encapsulant in an atmosphere of flowing hydrogen were studied in order to evaluate the distribution of tellurium concentration. No facets were formed in undoped GaSb, while in the case of Te-doped crystals the facets have appeared at the centre of the GaSb bowl. The formation of facets is dependent on the temperature conditions in the growth surface and on the level and the nonhomogeneity of dopant concentration near to the growth interface.

The quality of the crystals was characterized by X-ray topography. We employed the method of double crystal spectrometer in the parallel position and we used Bragg case geometry. Several types of GaSb crystals with different Te concentration were chosen for this study.

We understand by facet such a macroscopic part of the crystal, where the variations in the morphology of striations with respect to the nonfaceted region are observable. Parallel growth striations indicate a planar solid/liquid interface in the facet, contrary to the circular ones in the nonfaceted region. Facets in this sense display many times higher Te concentration and different character of etched surface with respect to the nonfaceted region. The higher concentration of Te in certain range causes the change from p- to n-type conductivity. The origin of the facets could be explained by poor stirring conditions of the melt near the center of growth rotation and/or by step by step growth mechanism from the supercooled melt at the center of rotation in contrast to the continuous growth at the periphery. The sharp facet boundary and the growth striation indicate that the latter reason is more relevant.

The distribution of carrier concentration in the portion of GaSb crystals where the interface between p- and n- type conductivity has occurred was examined and the interpretation of the inhomogeneities observed was suggested.

X-RAY DIFFRACTION INVESTIGATION OF THE PECULARITIES OF THE ELASTIC STRAIN RELAXATION IN LATTICE MISMATCHED HETEROSTRUCTURES GROWN BY MOCVD

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High resolution X-ray diffraction interference and differential methods were used for the characterization of epitaxial growth and elastic strain relaxation processes in InGaAs and (AlGa)InAs epitaxial layers grown by MOCVD technique. X-ray diffraction investigations were made by using a SEIFERT XRD 3000 HR system equipped with a monochromator for the primary side (2 Ge channel-cut crystals in (+,-,-,+) configuration, 220 reflection) and for the secondary side (flat Ge(220) analyzer crystal). The samples investigated were grown on precisely oriented GaAs(001) substrates. The InGaAs/GaAs double heterostructures represented $In_{0.2}Ga_{0.8}As$ quantum wells of various thickness from 8 nm up to 31 nm and cap GaAs layer. The other kind of investigated samples were $(Al_{0.5}Ga_{0.5})_{0.82}In_{0.18}As$ epitaxial layers with thickness from 100 nm up to 400 nm grown either immediately on GaAs substrate (type I) or on the 2 μm $Al_{0.55}Ga_{0.45}As$ buffer layer (type II).

The results of the investigation of InGaAs QW are that the process of elastic strain relaxation leads to the appearence of different structural defects which change the growth conditions and elastic strain in the heterostructure. The strong correlation between InGaAs strained layer thickness, types and density of generated defects, degree of elastic strain relaxation in heterostructures was shown, too.

For (AlGa)InAs/GaAs heterostructures the process of elastic strain relaxation in structures of type I leads to the common type of relaxed crystal structures with homogeneously distributed dislocation networks. The investigation of the second type of heterostructures shows that another type of relaxed crystal structures appears. This means that during the process of relaxation in elastically strained epitaxial layers two areas with significantly different extent of relaxation arise. In strained layer there is a "block" crystal structure which consists of rectangular blocks (size about 10-12 μm) with low density of dislocation networks (about $10^5/cm^2$) near the interface with (AlGa)InAs. These blocks are separated by vertical dislocation walls oriented in-plane along {110} direction with extremely high dislocation density (up to $10^9/cm^2$). The increasing of the thickness of the (AlGa)InAs layer results in the increasing of dislocation density in the walls and hence to increasing of the elastic strain in those areas. It leads to the propagation of these dislocations into the bottom AlGaAs layer and hence to further decreasing of its crystal perfection. This allows to suggest the strong correlation between the type of elastic strain relaxation and the existence of buffer AlGaAs layer or, in general, between the process of elastic strain relaxation and initial stage of epitaxial growth. The result of the preliminary investigation of similar samples additionally containing (AlAs-GaAs)SL is that the critical thickness of the strained layer may be exceeded three to five times.

HIGH RESOLUTION X-RAY DIFFRACTION CHARACTERIZATION OF Ge/Si/GaAs HETEROSTRUCTURES

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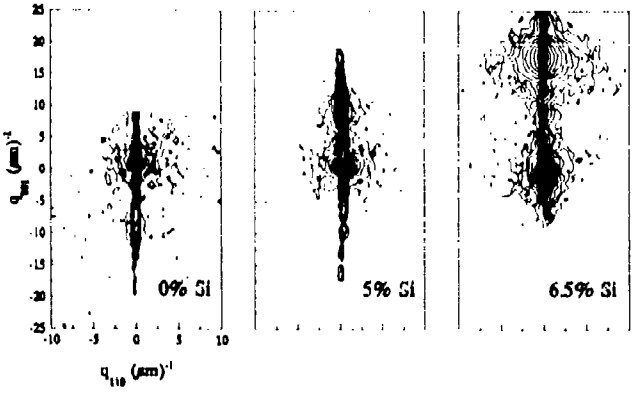
Heterostructures consisting of alloys of the Group IV elements Si and Ge with GaAs have attracted attention, since band structure calculations have predicted that the addition of Si to GaAs should increase the bandgap of dilute $(GaAs)_{1-x}(Si_2)_x$ alloys with the bandgap remaining direct up to $x \approx 0.2$. In $(GaAs)_{1-x}(Ge_2)_x$, the alloy is predicted to have a direct gap up to $x \approx 0.8$ while exhibiting a local minimum in the conduction band at $x \approx 0.3$. In order to achieve these properties, it is necessary to fabricate (GaAs)(Group IV) alloys without the potential degrading influences of lattice parameter mismatch, polar versus non-polar growth, and the prospect of order-disorder transformations. In the present work, we have used both double- and triple-crystal x-ray diffraction to study the structural characteristics of Si/Ge and Ge/GaAs alloys grown on GaAs substrates by molecular beam epitaxy (MBE). We have determined an optimized growth recipe for Ge/GaAs alloys in terms of four major growth parameters (substrate temperature, GaAs deposition rate, As_2 overpressure, and Ge mole fraction) by using the width of the alloy layer 004 reflection in a high resolution triple crystal transverse scan, which directly measures the range of angular misorientations in the layer independent of lattice dilations or strains. The optimized MBE growth environment was then used for the deposition of Ge-Si alloys with high structural quality on GaAs substrates, even though the growth of crystallographically perfect layers would be expected to be complicated by the diamond-on-zincblende materials system and the transition of the pseudomorphic strain from compressive to tensile with increasing Si mole fraction. The presence of well defined interference fringes in double crystal x-ray rocking curves showed a high level of structural perfection in layers with silicon mole fractions less than approximately 7.5%; subsequent increases in the silicon content led to an abrupt relaxation in the pseudomorphic strain in the SiGe alloy layers. Triple crystal diffraction scans from the same samples (see below) exhibited a continuous increase in the intensity of the diffuse scattering about the 004 reciprocal lattice point of the epitaxial layer, suggesting either the generation of dislocations or the nucleation of point defect clusters with increasing Si content. The physical origin of the diffuse scattering will be discussed.



(left) Triple crystal x-ray diffraction scans from Si/Ge alloy layers grown on GaAs; the 004 reciprocal lattice point from the GaAs substrate is centered in each illustration. The approximate Si mole fractions estimated from double crystal rocking curves are given with each scan.

A DIRECT OBTAINING OF STRAIN PROFILE FROM X-RAY ROCKING CURVES

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A study of multilayer crystals by X-ray high-resolution diffractometry proposes to obtain strain and damage depth profiles by the solving of the inverse problem of X-ray rocking curve interpretation. In this way the phase problem and numerical methods are severe. For the simultaneous solution these both problems in the discrete kinematical approach to the X-ray diffraction the method of Fourier coefficients (Stepanov, 1994) is presented. This method is based on the analysis of rocking curve Fourier coefficients and was earlier applied for graded layers (Stepanov, 1992; 1994) and three-layer laser heterostructures in the system AlGaAs (Stepanov, 1992) as well as for superlattices in the system SiGe (Stepanov & Liepinsh, 1993).

The method has some important advantages, so in the case of superlattices, it does not need the identification of satellite orders, if the superlattice period consists of N layers it allows to find 2N equivalent in some sense strain profiles. Using many rocking curves measured for different values of absorption coefficient it allows to reduce the inverse problem to the solving of linear algebraic system with Vandermonde's matrix. We note Petrashen & Chukhovskii, 1989, have solved the problem using two rocking curves and analytic continuation of them, as it is well known, the later problem is ill-posed one.

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ACCOUNTING FOR INTERFACIAL ROUGHNESS EFFECTS IN GRAZING-INCIDENCE X-RAY DIFFRACTION BY MULTILAYERS

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Recently, the grazing-incidence X-ray diffraction (GID) has been experimentally demonstrated to provide extensive informations on semiconductor multilayers. However, the quantitative interpretation of these data requires a proper theory.

In [1,2] a new theoretical description of GID in multilayers has been developed. In this method the solution of the extended dynamical diffraction equations was obtained in the form of a product of (4 × 4) characteristic matrices of the layers. In [2] this approach has been experimentally proved to be adequate for perfect multilayers.

Here, the extension of our matrix approach [1,2] is reported where the surface as well as uncorrelated interface roughness and transition layers in multilayers are taken into account.

The proposed theoretical approach is applicable for the simulation of effects of various multilayer defects on GID: roughness at interfaces between layers, fluctuations of layer thickness and density, partial disorder of crystalline structure, presence of transition layers etc.

It is proven by means of computer simulations that small interfacial roughness and thin transition layers in multilayers have a similar effect onto GID. In the case of superlattices (SL) the roughness and the transition layers result in the acceleration of the overall intensity drop with increasing glancing angles between X-rays and the surface and in the intensity decrease of high-order Sl peaks. The transition layer thickness or the rms roughness height can me measured due to these effects.

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ACCOUNTING FOR NORMAL LATTICE STRAIN EFFECT IN THE THEORY OF X-RAY DIFFRACTION UNDER TOTAL EXTERNAL REFLECTION CONDITIONS

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The actual depth scale in modern microelectronics has become as tiny as few atomic monolayers. That makes inapplicable conventional X-ray diffraction for controlling many technological processes since the extinction length in case X-ray diffraction is about 1-10μm. This problem has recently been overcome with the diffraction schemes where X-ray beams are grazing along the crystal surface. The extinction length is reduced there to 100nm-1μm due to the X-ray total external reflection effect.

The minimum extinction length is attained in the so-called grazing incidence diffraction (GID) scheme when both the incident and diffracted X-ray waves are grazing. Unfortunately, GID is often not helpful in measuring lattice strains in thin layers because the scattering vector of GID can partially lie in the surface and therefore the lateral lattice strain only can have effect on GID. However, the lateral strains in thin layers usually are accompanied by normal ones.

Thus the extremely asymmetric X-ray diffraction (XEAD) with the grazing incidence or grazing exit seems to be the most prospective technique for the studies of strains in surface layers because it combines the small extinction length with a possibility to measure normal lattice strain.

In the present contribution we report an approach for analysis of XEAD from single crystals and multilayers with normal lattice strain. The proposed algorithm is based on the extended dynamical diffraction theory with due account for the specular reflection and refraction effects. It is an extension of the recursion relation for GID in multilayers developed recently in [1,2]. This approach is also applicable to **asymmetric GID where both X-ray waves are grazing.**

The computed program based on the developed algorithm is used for the demonstration of the advantages of XEAD in studying lattice strains in thin layers. In particular, it is shown that in the symmetrical 220 reflection the maximum reflectivity of a strained *Ge* surface layer of 10 nm thickness ($\Delta a/a \sim 10^{-3}$) on a *Ge* crystal is only $2 \cdot 10^{-5}$. **Therefore the layer effect hardly would be detectable in the flank of the substrate Bragg peak. With XEAD the same layer may provide about ~ 20% of the substrate Bragg peak intensity in some non-coplanar cases.**

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AN ADVANCED METHOD FOR COMPUTATION OF X-RAY MULTIPLE BRAGG DIFFRACTION

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In recent years application of bright synchrotron radiation to X ray experiments provided an opportunity to measure fine structure of multiple Bragg diffraction peaks. The measurements can form the basis for new methods of studying crystals and their interfaces. However a proper theoretical interpretation is required.

The theory of [illegible] X-ray Bragg diffraction in perfect crystals [illegible] $2N \times 2N$ [illegible] The analytical solution [illegible] beams undergoing [illegible] $2N \times 2N$ [illegible] We [illegible] optimal [illegible]

Here we [illegible] X-ray Bragg diffraction [illegible] $2N \times N$ [illegible] N [illegible] The diffraction equations are presented in the form

$$\begin{pmatrix} \cdot & \cdot \\ \cdot & \cdot \end{pmatrix} \begin{pmatrix} \mathbf{D} \\ \mathbf{D} \end{pmatrix} = \begin{pmatrix} \cdot & \cdot \\ \cdot & \cdot \end{pmatrix} \begin{pmatrix} \mathbf{D} \\ \mathbf{D} \end{pmatrix},$$

where [illegible] and $\mathbf{D}$ are the [illegible] and the X ray wavefield amplitudes to be found as the eigenvalues and eigenvectors, respectively. Matrix $G^{ss'}_{hh'} = \alpha_h \delta^{ss'}_{hh'} +$ [illegible] $2N \times 2N$ scattering matrix, indexes h, h' enumerate the Bragg reflections and [illegible] de-rotate X ray polarizations; δ is the Kronecker symbol, α_h are the Bragg deviations parameters, $\chi_{\bar{h}}$ are the components of crystal dielectric susceptibility, $\mathbf{e}_s$ are the unit vectors along X ray polarizations; $2\Gamma^{ss'}_{hh'} = 2\gamma_h \delta^{ss'}_{hh'}$ is the diagonal matrix. Finally, P is the rectangular $2N \times 2N_S$ matrix:

$$P = \begin{pmatrix} \underbrace{\boxed{O}}_{2N_L} & \underbrace{\boxed{I}}_{2N_S} & \underbrace{\boxed{O}}_{2N_B} \end{pmatrix} \Big\} 2N \text{ rows},$$

where matrix I is the unit diagonal matrix, O are zero rectangular matrices, $\tilde{P}$ is the transposed matrix of P and N_L and N_B are the numbers of Bragg-case and Laue-case X-ray beams.

The proposed method has been implemented in a program and its validity has been proved with the test computations. In some cases the computation rate in our method is significantly faster than in [2] as the order of the matrices is proportional to the number of grazing beams whereas in [2] it is fixed.

We believe that our program can be successfully used in applications of X-ray multiple diffraction to crystal surface studies and in multi-beam optics of X-rays.

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X-RAY DIFFUSE SCATTERING FROM EPITAXIAL $CoSi_2/Si/CoSi_2$ LAYERS ON Si(111)

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Knowledge of the roughness parameters, i.e. the rms roughness, the in plane correlation function of the interfaces and their influence on each other (conformal roughness) is of practical interest. The fabrication of microelectronic devices demands for extremely thin metallic films with high electronic conductivity which is severely degraded by interfacial roughness.

X ray measurements of both the specular and diffuse scattering in the range of total external reflection were performed with a $CoSi_2/Si/CoSi_2$ Si(111) layer system produced by molecular beam epitaxy (MBE) /1/. The interpretation of the diffusely scattered intensity was done within the distorted wave Born approximation (DWBA) /2/ /3/, including the effect of conformal roughness /4/. Fits were performed on all measurements (specular, longitudinal diffuse, detector and transverse scans) simultaneously.

Conformal roughness of the interfaces of at least one of the thin $CoSi_2$ layers was found, whereas correlations between the interfaces of the buried Si layer could be excluded. Lateral correlation lengths are in the range of the average distance between two monolayer steps. The small magnitude of the roughness of the buried interfaces of this special sample, however, renders an exact determination of the lateral correlations very difficult.

The measurements were carried out using CuK_α radiation of an 18kW laboratory source and synchrotron radiation from the storage ring DORIS III at HASYLAB (Hamburg) on the wiggler beamline (station ROEWI) with a wavelength λ=1.659 Å.

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HIGH RESOLUTION SYNCHROTRON X-RAY DIFFRACTION TOMOGRAPHY OF POLYCRYSTALLINE SAMPLES

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In understanding the macroscopic response of polycrystalline structural materials to loading, it is frequently essential to know both the spatial distribution of strain and the variation of micro-texture. Most methods for mapping strain and for mapping micro-texture on a scale of 100 μm are intrinsically two-dimensional: samples must be serially sectioned to determine variations normal to a sample's surface. In the case of Al-Li 2090, for example, changes in the through-thickness texture are thought to be the reason this alloy exhibits fatigue crack propagation rates an order of magnitude better than those of other Al alloys. If one is to study the **evolution** of strain throughout such samples' lives, however, then a non-destructive, high resolution method of mapping in three-dimensions must be developed.

This paper describes several approaches used in developing high resolution synchrotron x-ray diffraction tomography of polycrystalline materials. Preliminary experiments at SSRL are reported on partially cracked compact tension samples of Al-Li 2090 and on model samples comprised of many randomly-packed, millimeter-sized pieces of Si wafers. Polychromatic beams collimated to 100 μm diameter have been used, and the results indicate that further collimation will not lead to prohibitive increases in data collection times. The distribution of diffracted intensity has been collected on high resolution x-ray film as well as on image storage plates, and, as the streaks from different hkl run together, Mo filters are placed in the incident beam to produce a large change in contrast on the detection medium at the position separating grains diffracting wavelengths just above and just below the K-edge. Micro-texture mapping in the compact tension samples is performed by translating the sample; different orientations of the incident beam are also used. Depth information is obtained by observing how the spatial distribution of diffracted intensity on the detection medium varies with changing sample to detector separation. The "polycrystalline" Si samples have been used to investigate how practical this approach is. Techniques for measuring the average strain within grains and for mapping the variation of strain as a function of three-dimensional position have also been investigated and will be discussed.

X-RAY DIFFUSE SCATTERING IN LANGMUIR-BLODGETT-MULTILAYERS PREPARED FROM FATTY ACID SALTS

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Multilayers from fatty acid salts can be prepared by the Langmuir- Blodgett technique. They exhibit a perfect lamellar stacking along the surface normal. The spacing is determined from the angular distance among the small angle Bragg peaks measured up to many orders using X-ray specular reflectivity. From the slope of intensity along q, the evaluated interface roughness between the metallic counter ions is of the order of one ionic diameter only. Along q, the films show a strong diffuse resonant scattering which varies with the periodicity of the multilayer. Its intensity depends on the scattering power of the respective counter ions and vanishes in case of pure acid films. Along q, we found a continous slope without resonant peaks but sharp Yoneda wings appear [1]. The curves can be described by a vertical correlation of 2-3 spacings and a lateral correlation length of the order of 1000Å using Sinha's approach[2] of the diffuse resonant scattering.

However, the in-plane structure of films is characterized by a powder like order. The amphiphilic molecules are organized within 2D domains having a diameter between 100Å and 1 µm surrounded by nonordered material. Among the domains the tilt of hydrocarbon chains is randomly distributed against the surface normal but it changes within the domains, too. Due to this real structure Sinha's model is inequate to describe the interface properties of our films. Therefore we describe the measured diffuse scattering by an other model. It respects the statistical dependence of the domain order and nearly vanished interface roughness. The respective results are compared with simulations performed in terms of Sinha's approach.

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DEPTH SENSITIVE X-RAY SCATTERING TOPOGRAPHIC OBSERVATION OF MBE GROWN InAs ON GaAs

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X-ray scattering topography, which the present authors proposed,[1-2] has been successfully applied to, lattice-mismatched heteroepitaxial layer systems, an MBE (molecular beam epitaxy) grown InAs on GaAs, MO-CVD (metal-organic chemical vapor deposition) grown GaAs on Si and InP on Si, of which systems have crystal mosicities which gave a local rocking curve of X-ray diffraction as broad as several hundreds arc sec. Since, for such a locally imperfect crystal, conventional X-ray diffraction topography (e.g. Lang-camera) provides little significant information, X-ray scattering topography has been applied to characterizing lattice-mismatched heteroepitaxial layer systems. Personal computer assisted X-ray scattering topography has enabled us to observe a quantitative orientation distribution.[3,4] And crystallographical correlation between the epitaxial layers and substrates have been also discussed.[5-7]

In this symposium we report the depth-sensitive X-ray scattering topographs of MBE grown InAs on GaAs, owing to different penetration depth of different diffraction index.

A Be-doped InAs layer was epitaxially grown to a thickness of 1.0 μm by MBE on Cr-O doped GaAs substrate which has a (001) surface orientation. The prepared layer has indeed two small local irregularities clearly observed in an optical micrograph, but otherwise it is perfectly mirrorlike. Throughout the X-ray experiments Co Kα radiation was used. Penetration depth of (004), (002) and (006) were calculated 0.6, 1.0, 5.8 μm, respectively, by dynamical theory. The experimental results present depth sensitivity depending on different diffraction index.

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HIGH-RESOLUTION STUDIES OF DOMAINS IN CRYSTALS OF THE $KTiOAsO_4$ FAMILY

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The aim of this investigation is to determine how crystals of the potassium titanyl arsenate, $KTiOAsO_4$, family can be optimized for application in nonlinear optical devices.

$KTiOAsO_4$ (KTA) is an analogue of the nonlinear optical crystal $KTiOPO_4$ (KTP) which is widely used in second-harmonic generation (SHG) from Nd:YAG radiation at 1.06 μm. The initial work[1] on KTA suggested that it was superior to KTP for SHG from Nd:YAG laser sources. However, subsequent workers[2] could not reproduce these results. These later failures to achieve the expected performance were thought to result from the presence of 180 ° (polar) domains in as-grown KTA. Some controversy raged about this for a number of years with evidence for and against the presence of domains being reported. Eventually, the original workers[3] reported that their results had been obtained on crystals unintentionally doped with Fe^{3+}. Although this accounted for the unusual efficiency of their KTA samples, much confusion about the nature of the domain-structure in KTA crystals grown from different fluxes in the presence of different dopants such as In and Fe, remains today.

In order to resolve the current confusion about these crystals, high-resolution diffraction (rocking curves and reciprocal space mapping) and topography are being employed to investigate both domain formation in particular and the quality of the crystals in general. The question of whether or not polar domains are present is addressed by the choice of reflections that are particularly sensitive to anomalous scattering at the CuKα wavelength. The instrument used is the Philips HRD employing a 4-bounce Bartels monochromator set for Ge 220 with triple-axis attachment as and when required. The results obtained so far are presented here and discussed in the context of the optical performance of these materials.

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X-RAY INTERFEROMETRIC COMPUTERIZED TOMOGRAPHY

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The possibility of diagnostics of weak deformations of monocrystals by means of computerized tomography (CT) based on the phenomenon of dynamical diffraction of hard X-rays
on crystals was considered. Having this aim in view, a geometrical optics of dynamical diffraction of X-rays in crystals was developed in the analogy with [1], but in the approximation of rectilinear trajectories. This essentially simplified mathematical treatment of the problem and allowed to apply early developed CT algorithms to other interactions of radiation with a sample.

In the proposed scheme a spherical X-ray wave is incident on crystalline plate oriented according to Laue case. In X-rays reflected from atomic planes of the crystal, there arise interference pattern (anomalous Pendellosung fringes [2]) which is sensitive to crystal deformation field.

As initial data for CT serve a series of above Pendellosung fringes obtained at different positions of X-ray source. After the calculation of space distributions of phase differences in interfering beams a reconstruction of the initial deformation field is made by using the CT procedure of finite series expansion, witch was slightly modified based on the features of our scheme. A computerized simulation of the experiment and further restoration of crystal deformation field showed sufficient accuracy and stability of the proposed technique against imminent experimental errors of different types.

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X-RAY GEOMETRICAL OPTICS IN RECTILINEAR PATH APPROXIMATION

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The geometrical optics of X-ray diffraction in distorted crystals was developed in [1,2]. Our derivation is analogous to that in many respects except for the fact, that in our case the eikonal of diffracted rays is independent of the crystal deformation field. In this approximation the trajectories of diffracted rays are rectilinear and are independent from the displacement vector of the deformation field of crystals, of course, at the expanse of introduction of more rigid restrictions on crystal deformations. However, these restrictions were assessed to allow for deformations sufficient for essential change of diffracted beams phases and those for interference pattern obtained from the crystal under investigation. We have familiar ideal crystal solutions with additional phase factor due to the crystal deformation. The paths of rays as well as their amplitudes are similar to those for the ideal crystal, the additional phases equal to the integral along the ray paths and depends from displacement fields. Calculations in geometrical optics approximation and by numerical integration of Takagi's equations are compared.

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TOPOGRAPHY METHOD OF QUANTITATIVE STRUCTURE INVESTIGATION OF SUPERLATTICES AND DISTORTED CRYSTALS

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Even the most perfect semiconductor superlattices (SL) are usually grown with SL period inhomogeneity of the order of 1% across the surface of a wafer. Because of this, the development of the quantitative X-ray topography method, known for imperfect crystals [1,2], becomes possible. Such a method is presented in the report for the first time.

In [1,2] the information results from the investigation of behavior of the diffraction contours in topographs. For topography measurement of an imperfect crystal, the use of a single reflection can be sufficient. For SL, at least 3 reflections (the substrate peak and two SL satellites) must be used. The method proposed can work for observation and calculation of inhomogeneity of 3 types of SL parameters across the surface of the wafer. These parameters are the SL average composition, taken within the SL thickness, the SL period, and the SL orientation. For application of the method, the high-intensity X ray source is required. The Synchrotron irradiation equipment is the most suitable.

It is advantageous to analyse SL inhomogeneity in two stages. Firstly, the qualitatively observation of SL structure is necessary. A search for several surface areas, at which the diffraction contours are collapsed [2], is desirable. For these areas, the inhomogeneity of structure parameters is revealed to be often maximum. The second stage of the analys is calculation procedure at areas with maximum inhomogeneity and at predetermined areas. The examples of investigations for volume crystal and SL structure parameters by the proposed method are presented.

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SYNCHROTRON DIFFRACTION TOPOGRAPHY ANALYSIS OF AlAs/AlGaAs SUPERLATTICE

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Results of X-ray diffraction topography investigations of the AlAs/$Al_{0.2}Ga_{0.8}As$ superlattice (SL) grown by MBE method on the (100) GaAs substrate are presented. For topography, the 400 GaAs reflection as well as the SL satellites: -1, 0, +1 and +3 situated in the neighborhood of this substrate reflection were used. To enhance the intensities of satellite peaks the equipment of Siberian Synchrotron Irradiation Center was employed.

A high irradiation intensity permitted to registrate the satellite reflection topographs directly at the fluorescence screen. These "screen" satellite topographs gave possibility to observe the diffraction contours moved with specimen rotation around the Bragg axis. In topographs, recorded using different satellites, contours moved at various directions and with various velocities. By going across the surface of the specimen, the observed picture was considerably affected. These effects allowed to choose the area of SL with maximum distortions for subsequent analysis and measurement procedure using the proposed method (see abstract by Trukhanov in this issue).

To calculate the SL distortions the series of topographs were registrated on photoplates using the substrate reflection and the mentioned satellites. Topographs showing the contour behavior for different satellites are presented in the report. The values of maximum variations of SL period (1.9 nm) and of average SL composition ($8,6x10^{-3}$ at.fraction) have been calculated. The values of SL parameters were 148 nm for SL period and 0.65 at.fraction of Al for average composition.

ACCOUNTING FOR CORRELATED ROUGHNESS EFFECTS IN X-RAY GRAZING-INCIDENCE DIFFRACTION

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Recently the X-ray grazing-incidence diffraction method (GID) has been effectively applied to the studies of synthetic multilayers (see, for example, [1]). It has been shown experimentally that various kinds of defects in multilayers can be distinctly measured with GID. However, there is a lack of theoretical models. We believe that a new matrix approach for description of GID in multilayers proposed in [2] can be used as a basis for these models. In particular this approach has been extended for uncorrelated interface roughness in multilayers [3].

In the present report we propose an advanced theory which takes into account the effects of correlated roughness. Our method provides the computation of GID intensities with respect to the rms interfacial roughness, height-height correlation length and amplitude.

The GID intensities are calculated in the dynamical diffraction approximation and the corrections due to roughness are calculated in the first Born approximation. The total scattering matrix has the form:

$$\left\langle \hat{S} \right\rangle^{corr} = \left\langle \hat{S} \right\rangle \left\{ 1 + \frac{1}{2} \sum_{\mu \neq \nu} g_{\mu\nu} (u^{\mu-1} - u^{\mu})(u^{\nu-1} - u^{\nu}) k_0^2 \sigma_\mu \sigma_\nu + \frac{1}{4} \sum_{\mu \neq \nu} g_{\mu\nu}^2 \right\},$$

where $\left\langle \hat{S} \right\rangle$ is the final scattering matrix for multilayer structure with uncorrelated interfacial roughness as in [3], u^μ are the roots of the dispersion equation of dynamical diffraction in the μ-th layer; k_0 is the module of the X-ray wave vector in vacuum; σ_μ is the rms roughness height for μ-th interface; $g_{\mu\nu}$ is the nondiagonal matrix element of correlator matrix: $g_{\mu\nu} = v_A \exp\{-|Z_\mu - Z_\nu| / v_l\}$. The parameters v_A and v_l are the amplitude and length of correlations, respectively; Z_μ is the coordinate of μ-th interface. The limits of validity of the Born approximation are discussed.

The computations carried out with the developed computer program show that GID can be employed as convenient tool for nondestructive characterization of epilayers thickness and interfacial roughness and height-height correlations between microstructures of different interfaces in multilayers and superlattices.

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DETERMINATION OF THE LATTICE RELAXATION OF NANOSTRUCTURED SIGE/SI PILLARS BY HIGH-RESOLUTION X-RAY DIFFRACTION

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Two-dimensional periodic arrays of pillars have gained much interest in view of the fabrication of nanoscale features in Si [1]. We have made $Si_{1-x}Ge_x$ pillars by etching an (001) oriented Si wafer epitaxially overgrown with $Si_{1-x}Ge_x$. The pillars were etched to such a depth that the lower part consists of Si and the upper part (top) of $Si_{1-x}Ge_x$.

We employed high-resolution X-ray diffraction to obtain the **periodicity** and **shape** of the pillars and the **lattice parameters** in the pillars. A Philips high-resolution diffractometer (HR-1) was used with four Ge (220) reflections in the monochromator, tuned to the CuKα1 line at 0.15406 nm. For the two-dimensional mapping a receiving slit was used in front of the detector to obtain enhanced resolution in the diffraction angle.

Two-dimensional maps near the 113 and 224 reciprocal lattice points of two-dimensional periodic arrays of small (<250 nm) semiconductor pillars were measured and compared with kinematical diffraction-model calculations [2]. These are based on Fourier transformation of the Si and $Si_{1-x}Ge_x$ parts of the grating separately.

The periodicity of the grating is obtained from the distance of the satellite peaks close to the Bragg peak that originate from the three-dimensional periodicity of the crystal lattice.

The thickness of the Si and $Si_{1-x}Ge_x$ parts and width of the pillars are determined by matching the intensity of the individual satellites and the intensity ratio of the Si and $Si_{1-x}Ge_x$ parts of the diffraction pattern.

We will show that complete lattice relaxation occurs in the small (200x200 nm^2) pillars, whereas the lattice is fully strained in large (10x10 mm^2) pillars.

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DYNAMICAL THEORY APPLIED TO CRYSTALS WITH A STATISTICALLY DEFORMED LAYER.

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The statistical dynamical theory dealing with an homogeneous distribution of microdefects was first proposed by Kato (1). Later, the angular distribution of the diffracted intensity was considered in (2).

X-ray intensity diffracted by statistically deformed crystals is usually considered as the sum of a coherent I_{coh} and an incoherent I_{inc} components, which can be separated experimentally by using the technique of triple-crystal diffractometry (3). The present work deals with numerical calculations of the angular distribution of I_{coh} from a crystal with a statistical distribution of microdefects located in a surface layer, in the frame of the theory developed in (2). The lattice spacing of the layer was taken slightly different from the lattice spacing of the perfect substrate (such a situation can appear for instance in the case of epitaxial growth). The statistical layer deformation is defined, according to (1) by a static Debye-Waller factor E and a correlation function $g(\xi)$. We have used different models for $g(\xi)$, an exponential model $g(\xi)=\exp(-|\xi|/\tau)$ and a function corresponding to spherical amorphous clusters. In our calculations of the coherent intensity, I^s_{coh} diffracted by the substrate and I^l_{coh} diffracted by the layer correspond to different angular ranges because of the macroscopic deformation of the layer. Our results show that I^l_{coh} depends strongly on the value of E and on the value of the correlation length $\tau=\int_0^\infty dx\ g(\xi)$ only and practically does not depend on the model of correlation function. The strong dependence on τ gives opportunity to determine τ directly from experiment if E is known. On the contrary the behavior of I^s_{coh} appears to be strongly dependent on the form of the correlation function $g(\xi)$.

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X-RAY STANDING WAVES AND HIGH PRECISION X-RAY DIFFRACTION IN REAL CRYSTALS. THEORY AND APPLICATIONS.

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The X-ray Standing Wave (XSW) method now is a powerful tool for determining the structure of surfaces and the position of adsorbed atoms on the top of the crystals. In the most of applications of XSW method dynamical theory of X-ray propagation in a perfect crystal is used. However this method can be successfully applied also for investigation of the structure of the real crystals for e.g. crystals with deformed surface layers. It gives also an opportunity to determine the position of the impurity atoms in that structures. General theory of XSW method in real crystals is presented. The possibilities of the method are demonstrated on several examples. Investigation of the implanted crystals: structure of Si crystals implanted by Fe and Ni atoms. Investigation of artificial nanometer-scale materials: structure of $(InAs)_1(GaAs)_1$ layered crystals. Combination of XSW and High Precision Diffraction methods opens the possibilities for phase analysis and gives an opportunity for the unique determination of the profile of deformation of the crystal surface. Theoretical results for investigation of the bent crystals by XSW method are also presented. There were obtained angular dependencies for the secondary radiation yield (photoemission and fluorescence) in such crystals. The behaviour of the wavefields in the volume of the curved crystal were also investigated.

THE DETERMINATION OF QUASI-PLASTIC STRAINS IN A CRYSTAL PLATE BY THE SOLUTION OF THE INVERSE PROBLEM OF THE ELASTICITY THEORY (A ONE-DIMENSIONAL CASE).

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A series of plane wave topographs can be used for the reconstructing of distortion field in a crystal plate. In the case of a weak distortion it components w_{ij} can be calculated in the form of halftone images from the system of linear equations using original topographic images [1].

The w_{ij} obtained provide the determination of a one-dimensional distribution of a predominant impurity in the plate. Following authors of [2] one can obtain the solution by the backward calculations using just one of the w_{ij}. Analysing the solution we have found that the error can reach a large value especially for materials with a high Poison's ratio. In some cases, when the transfer function for the chosen w_{ij} tends to zero the solution may be fully incorrect.

The consideration of the linearized inverse problem of the elasticity theory provides the solution with a good accuracy if the elastic strain gradient is quite low. In present work the problem has been solved for the case of anisotropy both of the crystal and point defect properties and under assumption that the distortions are measured on a small (but non a zero) distance from the sample surface. The crystal plate has at least one elastic symmetry plane parallel to the crystal surface then the function of the impurity distribution $f(x)$ can be represented as a linear combination of at most four measured distortion components

$$f(x) = \sum_{i,j} l_{ij} w_{ij}(x)$$

In a fully orthotropic case it requires of only two w_{ij}. The coefficients l_{ij} of the combination are the functions of the elastic properties of the material and impurity. Such a representation of $f(x)$ is right for any point of a plate but just near the plate surface the error of the solution is most low. The later condition is suitable to the X-ray topographs made in Bragg geometry. Due to the simplicity of the solution form it also can be applied to local detecting of impurity concentration by means of X-ray diffractometry techniques.

The estimating of the solution accuracy indicates that the error is of the order of $(\tau/\lambda)^2$ (here λ is a period of impurity inhomogeneity, τ - a distance from the plate surface) and decrease with increasing of Poison's ratio.

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APPLICATIONS OF X-RAY TOPOGRAPHY OF SILICON WAFERS IN AN INDUSTRIAL LABORATORY

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The IBM Analysis Service supports internal (e.g. 4 MBit line in Böblingen) and external customers in a wide spectrum of physical, chemical and metrological methods. Here, typical applications of Single Crystal X-ray Topography are presented to demonstrate the usefulness and benefits in supporting silicon device fabrication.

The RIGAKU LGL-8 Lang Camera with a 18 kW rotating anode, an on-line imaging and warpage correction system is used for rapid analysis of wafers up to 200mm.

Dicing Problem: severe edge chipping and rapid blade wear out occurred during dicing the wafers. First, the reason was believed to be a new charge of blades from a new supplier. But Topography could quickly reveal heavy crystal damage as the responsible factor. Analysis of wafers after the most critical hot processes identified HIPOX (High Pressure Oxidation) as the source of the high dislocation density.
The reason was a 4 times higher take out speed from an oven than normal. Thus, faster cooling at the wafer rim resulted in stresses exceeding the yield point in the middle and dislocations were punched out at the Si/SiO_2 interface (TEM)
Subsequently, the WIP (Work in Process) was inspected with the on-line camera system and the affected parts (lots) were routed to a "super smooth" dicing process.

Epi-Wafer Optimization: Two trends characterize today's manufacturing of p^-p^+ epitaxial wafers: a) Reduction of interstitial oxygen in the p^- bulk to increase the defect free zone under the epilayer. This results in lower intrinsic gettering efficiency which is counterbalanced by extrinsic gettering of defects introduced by backside sand blast.
b) Replacement of batch epi-reactors by single-wafer reactors to increase the thruput and to improve the epi thickness and carrier concentration profile. The first generation of these reactors is plagued by a high probability for slip generation.
X-ray topography demonstrates its versatility in detecting the above defects by transmission and cross sectional imaging. Especially in these cases of low defect density it is superior to etching methods.

THE NUMERICAL SIMULATION OF BRAGG-CASE TOPOGRAPHIC IMAGES OF DISLOCATIONS AND PRECIPITATES IN GAAS EPITAXIAL LAYERS

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The numerical integration of Takagi-Taupin equations was used for simulation of the back-reflection double-crystal images of crystallographic defects in GaAs epitaxial layers. In particular, the images of dislocations inclined to the surface, of point-like precipitates and misfit dislocation crossings were studied in aspect of their identification in practical examination of epitaxial layers.

The actual program used the formal assumption of the finite crystal thickness. That allowed us to use the integration grid with boundaries situated far from the defects, and less time consumable extension of the simulated area. The simulations took into account the diffraction effects both in the layer and in the substrate and diffused lattice parameter profiles. The divergence of the beam was into account by adding of 50-100 plane-wave images. That improved the reality of the simulations by dumping some of the interference fringes.

We found that the images of dislocations, especially those inclined to the surface, are dominated by the direct dilatation-orientation contrast. The similarity of the contrast coming from Takagi-Taupin theory and that given by Bonse's approximation was confirmed. The greatest amount of the interference effect was observed for the images of point-like defect. The difference of the images taken in equivalent points of the layer and substrate peak was relatively small for the dislocations inclined to the surface.

The theoretical images were compared with the experimental topographs of 4-5μm thick GaAs epitaxial layers grown with *MOCVD* technique. The GaAs substrates with low concentration of defects, topographically examined before the growth process, were used. A high perfection of the epitaxial layers was confirmed and most of the dislocation in the layers was found to be the continuation of those in the substrates. Several cases of good correspondence of simulated and experimental images of dislocations was found.

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THE TRANSMISSION DIFFRACTION PATTERNS OF SILICON IMPLANTED WITH HIGH ENERGY α-PARTICLES

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2 mm thick silicon wafers, implanted with 4-5 MeV α-particles are studied by means of transmission diffraction topography including single-crystal projection (i) and section (ii) methods and transmission double-crystal method (iii).

It was found that all the three methods produced a negligible contrast in the symmetric transmission reflection, apart from some fragments of the implanted-area boundaries. That points to the continuity of the lattice planes and the unchanged interplanar distance in the case of lattice planes perpendicular to the surface of the sample.

The distinct interference fringes were observed with all the three methods in the case of asymmetric reflections. The appearance of the fringes in different methods and in different reflections was in general agreement with the prediction of the Bonse-Hart model (U. Bonse, M. Hart: phys. stat. sol. (a) 33, (1969), 351), but some features of the topographic image, requiring more complicated models, were found. In particular, the section topographs exhibited the curvature of the fringes, which may be interpreted as due to the change of the implanted ion dose along the beam intersecting the crystal.

The simulations of section and double-crystal topographic contrast were obtained using a program based on the numerical integration of the Takagi-Taupin equations. The deformation field, confirmed by our previous investigations in the back-reflection geometry was assumed (K. Wieteska, W. Wierzchowski: paper submitted to physica status solidi (a)). That assumed a gradual change of the interplanar distance of the planes parallel to the surface, according to the function obtained using Biersack-Ziegler TRIM-85 program or approximated by a fragment of hyperbola.

CHARACTERIZATION AND RELAXATION OF II-VI-EPITAXIAL SEMICONDUCTOR LAYERS BY HIGH RESOLUTION X-RAY DIFFRACTION

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The width of a rocking-curve (FWHM) is often used as an indicator for the crystalline perfection of a layer. Rocking-curve measurements also represent an efficiant tool to provide the perpendicular and the in-plane lattice mismatch for the determination of strain [1] and for observing of a relaxation process. For our measurements we constructed and used an (n,-n, m)-three-crystal-arrangement to investigate ZnTe- and HgSe/ZnTe-layers, grown on a [001]-orientated GaAs-substrate as well with molecular-beam-epitaxy (MBE) as with hot-wall-epitaxy (HWE). Our investigations of this semiconductor layers yielded a dependency between FWHM and layer thickness. Because of the large lattice mismatch between ZnTe and GaAs, we used the relations of van der Sluis [2] which are generell accurate, also for this hetero-structures to get the perpendicular lattice mismatch and observed the dependency between relaxation process and layer thickness. We also compared the different samples grown with MBE and HWE.

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HRXRD Investigations on MOVPE and MBE grown ZnSe/GaAs and $ZnSe_xTe_{1-x}$/GaAs Layers

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Key words: HRXRD, ZnSe, ZnSeTe

Single crystalline ZnSe, and $ZnSe_xTe_{1-x}$ layers were grown by metalorganic vapor phase epitaxy (MOVPE) on (001) GaAs substrates. The lattice mismatch between epi-layer and substrate is -0.27% for ZnSe/GaAs at a growth temperature of 340°C. The corresponding strain can be relaxed by the nucleation of misfit dislocations. The crystalline quality and the residual strain of the disturbed epi-layer were investigated by measuring rocking curves with a high resolution X-ray diffractometer consisting of a four reflection monochromator and a two reflection analyzer crystal. The two reflection crystal in front of the detector for analyzing the scattered beam enables the determination of the scattering vector $\vec{q}$ by measuring the reflected intensity near reciporcal lattice points (RLP). These $\vec{q}$-scan maps allow to distinguish effects of mosaicity, relaxation and variations of lattice plane distances. Different kind of disturbances lead to a broadening of the intensity distribution near a RLP in different directions in reciprocal space. A mosaic structure of the layer causes a broadening of the $\vec{q}$-scan intensity perpendicular to the according reciprocal lattice vector (RLV), whereas a variation of lattice plane distances leads to a broadening in the parallel direction.

For ZnSe/GaAs layers, the FWHM-values of $\vec{q}$-scan maps perpendicular to the direction of the RGP depends on the degree of strain relaxation in the layer. A critical thickness of 220±20nm and 100±20nm for MBE and MOVPE grown ZnSe/GaAs-layers respectively was found. In the direction parallel to the RLV, the FWHM-values are determined by inhomogenities of the strain relaxation, which depend on the growth conditions. The FWHM-values of rocking curves for the (004)-reflection, which are measured without analyzer crystal and the FWHM-values of $\vec{q}$-scan maps perpendicular and parallel to the RLV are independent from the growth method for relaxed layers with d>1μm (300", 250" and 80").

Misfit dislocations can be devided into an interfacial segment at the layer-substrate-interface and into a threading segment, which penetrates the hole layer. The density of the interfacial segments at the interface is determined by the strain relaxation, and therefore of the same value for relaxed MBE- and MOVPE-layers with d>1μm. However 'Plan-view'-TEM-pictures show a higher density of threading segments for MOVPE-layers compared to MBE-layers of the same strain relaxation. In HRXRD-measurements, the same FWHM-values of (004)-rocking curves for relaxed layers are observed. Obviously, the broadening of the scattered X-ray intensity for the (004)-reflection is mainly influenced by interfacial segments.

For $ZnSe_xTe_{1-x}$ layers with compositions of $0.2 < x < 0.8$ grown on GaAs(001) a spontaneous ordering (CuPt-strcture) is observed similar to III-V-alloys as $Ga_xIn_{1-x}P$. This ordered structure causes a weak signal of a $(\frac{1}{2}\frac{1}{2}\frac{1}{2})$-reflection, which is forbidden for a random distribution of Se- and Te-atoms.

FIRST EXPERIENCE WITH HIGH RESOLUTION DIFFRACTION EXPERIMENTS ON BEAMLINE 10 AT THE ESRF

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In April 1994 the first high resolution diffraction experiments were possible at the three axes diffractometer on beamline 10 (optics beamline) at the ESRF

The X-ray source of this beamline (BL) is a bending magnet with a source size of about 0.38 x 0.30 mm^2 at a distance of 40 m with a total power of 224 W at 100 mA. The reflection plane of the diffractometer is horizontal, following the π-polarization component can be used only. Since at that time the BL did not contain any optical elements, the first two axes of the diffractometer were used to realize a 220 double crystal monochromator. An energy of 8.05 keV was selected to have a comparison to measurements at conventional X-ray generators with CuK_α-radiation.

We present the results of measurements at 100 oriented Si wafers with pseudomorphic SiGe layers of different thickness and Ge content, SiGe multiple quantum well structures, and very thin (4 µm and 1 µm) Si crystals. The 400-rocking curves obtained at BL10 are compared to similar curves measured at a standard double crystal diffractometer in (n,-n) setting, and at a so-called high resolution diffractometer with Bartels monochromator with and without channel-cut analyzer.

Despite of different not optimal factors (3 reflections with π-polarization, long beam path in air with relatively strong absorption, dispersive (n,-n,m) arrangement, etc.) an intensity gain of more than two orders in comparison to typical parameters of conventional X-ray sources and diffractometers was obtained. The diffractometer and the X-ray beam are very stable. Due to the low horizontal and vertical beam divergence no special efforts are necessary to adjust the sample tilt and no significant broadening of the rocking curve of the Si substrates caused by the dispersive arrangement was observed. The rocking curves show an excellent signal-to-noise ratio. This is especially demonstrated in much deeper minima of the intensity oscillations of layer structures.

These first experiments showed that BL10 at ESRF will be a very powerful instrument for high resolution diffraction experiments especially after further improvements of the optics in front of the diffractometer.

INVESTIGATION OF STRAINED INGAAS/GAAS QUANTUM WELLS BY HIGH RESOLUTION X-RAY DIFFRACTION

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Thin strained InGaAs-layers are widely used to design wavelength and electrical properties of semiconductor lasers and transistors on the basis of GaAs.
These properties are determined by the shape of the quantum well (QW) itself, i.e. its depth, which is given by the In-content, thickness and a possible grading of the interfaces.
Furthermore the possibility of strain relaxation has to be taken into accout depending on the growth parameters (deposition time, III/V-ratio and substrate temperature) and the total strain. High resolution x-ray diffraction (HRXRD) was applied to investigate single QWs with different In-content sandwiched between GaAs using a diffractometer equipped with a four crystal monochromator.
The degree of relaxation can be evaluated by measuring the lattice parameters parallel and perpendicular to the (001) sample surface using (004) and (115) reflections.
Using the triple axis geometry of the diffractometer the reciprocal space was mapped around the (004) reflection. Then the sample was rotated by 90° and the two area scans were compared. If the sample is fully strained no difference is observed between both maps. In the case of partial relaxation one of the maps shows a shift of the layer peak in the q_x-direction of the reciprocal space with respect to the layer peak, whereas the shift disappears in the 90° rotated map.
The onset of relaxation by misfit dislocations is also detected in cathodoluminescence. In samples with a low degree of relaxation dark line defects (DLDs) are observed in one [110]-direction only. This points to a monoclinic distortion of the lattice even in the initial state of relaxation. That explanation is in accordance with the area maps in XRD.
The experimental (004) rocking curves were compared with theoretical ones using a simulation with the Takagi-Taupin formalism. InGaAs-layer thickness d and InAs-content x were determined from the best fit between the two curves. With these results the band structure of the QW can be calculated and the obtained theoretical values for the wavelength are compared to the measured PL-wavelength.
For smaller layer thicknesses it was possible to simulate the rocking curve by assuming sharp QW-interfaces. For thicker QWs a grading on the upper interface has to be included to allow for a good fit of the measured rocking curves. The grading is caused by In-segregation on InGaAs and subsequent incorporation into the next GaAs layer. The thickness of this graded layer can be assessed using synchrotron X-ray grazing incidence diffraction. From the simulation of the obtained curves the thickness of the graded layer has been estimated to be ≤ 1 nm for a 12.5 nm thick QW ($x = 0.18$).

UNIVERSAL ANALYTICAL TREATMENT FOR MANY-WAVE X-RAYS DIFFRACTION IN CRYSTAL PLATE

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Kravchenko str.,8, Moscow, 117331 Russia

In frame of dynamical diffraction theory [1] simple universal analytical solution of problem of N-wave diffraction of X-rays in crystal plate of arbitrary thickness is obtained. The results can be used for effective direct numerical calculations of characteristics of mani-wave diffraction.

1. S.L. Chang, Multiple Diffraction of X-Rays in Crystals, Springer-Verlag, 1984

SOME CRITERIA FOR MINIMIZING THE HEAT LOAD PROBLEM IN WHITE BEAM TOPOGRAPHY.

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European Synchrotron Radiation Facility, B.P. 220 Grenoble, France

The major drawback when working with very intense beams like those delivered by third generation synchrotron radiation machines is the huge amount of power impinging on the first element encountered by the beam. This is extremely crucial for the white beam topography technique, since the first element is the sample itself. The large beams normally used for topography may reach power loads of 10 W/mm^2 or more at normal incidence, and may easily introduce some non negligible thermal distortions that make the topographs meaningless, or even destroy the sample. Usually the first effect that appears in the topographs is some contrast associated with the thermally distorted area of the sample. Depending on the type of material, i.e. conductor or insulator, some criteria are derived for both cases in order to minimize such undesired distortions of the crystal. These concepts are also important for monochromators which have to deliver large homogeneous beams. One of the solutions to avoid complicated cooling geometries is to use thin crystals
Topographical and diffractometrical measurements were carried out in order to determine the thermal distortion and the average temperature gradient for different filtering conditions. This allows the mathematical model used for the criteria to be checked and the feasibility of the "thin crystal" option as a possible hard X-ray monochromator to be determined.

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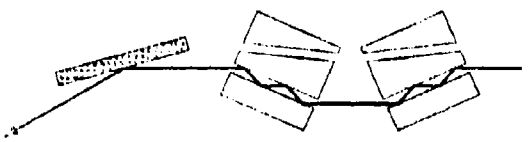
D³system

The State of the Art High Resolution X-ray Characterisation System

Bede scientific

Advanced Monochromator Design



High resolution mode with 8 bounces gives
>50 000 cps at 4.4" divergence, $\Delta\lambda/\lambda = 4.9 \times 10^{-5}$

High intensity mode with 4 asymmetric bounces gives
>400 000 cps at 11.5" divergence, $\Delta\lambda/\lambda = 1.4 \times 10^{-4}$

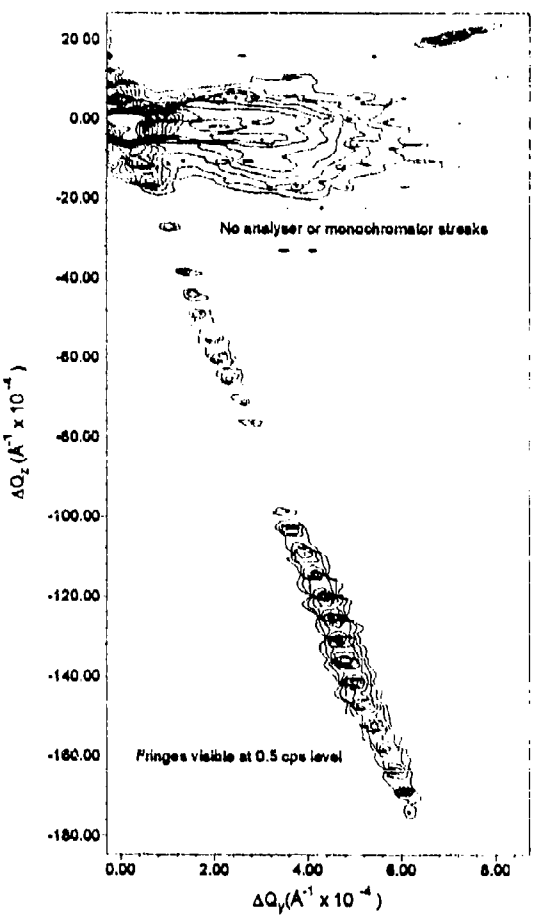
Flexibility

Precision

Accuracy

Speed

The Cleanest Reciprocal Space Maps



High Resolution triple axis reciprocal space map of a HEMT device, showing layers of good quality but tilted with respect to the substrate.

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